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Article

# Physical, Chemical, and Mechanical Characterization of Okra (*Abelmoschus Esculentus*) Fibers from the Littoral Region of Cameroon for Composite

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## Abstract

Okra (*Abelmoschus esculentus*) stems constitute an abundant lignocellulosic biomass with significant potential for sustainable composite reinforcement. In this study, okra fibers were extracted using biological retting, alkaline treatment (1-7.5 wt% NaOH), and combined extraction processes. The influence of extraction conditions on the physicochemical, mechanical, thermal, and structural properties of the fibers was investigated. FTIR analysis revealed the progressive removal of hemicellulose and lignin after alkaline treatment, while XRD results showed an increase in cellulose crystallinity. Optical microscopy observations revealed progressive fiber separation and cleaner surface morphology after alkaline treatment. Fiber density increased with NaOH concentration, whereas water absorption and moisture regain decreased due to the reduction of hydrophilic amorphous components. Mechanical properties, particularly tensile strength and Young's modulus, improved under moderate treatment conditions but decreased under severe alkaline conditions because of partial cellulose degradation. The optimal treatment condition (1 wt% NaOH for 60 min) provided the best balance between delignification, structural preservation, and mechanical performance. These results demonstrate that okra fibers are promising lightweight reinforcements for sustainable bio-composite and technical textile applications.

**Keywords:** okra fibers; *Abelmoschus esculentus*; alkali treatment; natural fibers; bio-composites; fiber extraction

## 1. Introduction

In the context of escalating environmental concerns and the progressive depletion of fossil-based resources, the development of sustainable and bio-based materials has become a central scientific and industrial priority [1]. Natural lignocellulosic fibers have emerged as promising alternatives to synthetic reinforcements due to their biodegradability, renewability, low cost, and relatively low density, typically ranging between 1.2 and 1.5 g·cm<sup>-3</sup> [2]. These fibers are increasingly employed in a wide range of applications, including textiles, automotive components, building materials, and lightweight composite structures, where they contribute to reducing carbon footprint while maintaining acceptable mechanical performance [3].

However, despite these advantages, the large variability in the properties of natural fibers remains a major limitation to their widespread industrial adoption. This variability is primarily attributed to differences in botanical origin, plant maturity, climatic conditions, and processing methods [4]. In biomass-rich regions such as Central Africa, the valorization of underexploited lignocellulosic resources represents a significant opportunity for sustainable development and circular economy strategies [5]. Cameroon, in particular, possesses abundant plant residues that

remain insufficiently exploited despite their promising characteristics [6]. Previous studies have demonstrated the potential of several local fibers, including banana, raffia, luffa, and *Sida rhombi folia*, for textile and composite applications [7], with reported improvements in mechanical behavior and physicochemical stability [8]. Among these resources, okra (*Abelmoschus esculentus*) is widely cultivated for its edible pods, generating substantial quantities of lignocellulosic stem residues after harvesting [9]. These stems contain bast fibers that are structurally comparable to other commercially exploited fibers derived from plant bark or pseudo stems [10]. Nevertheless, okra fibers remain poorly studied, particularly in terms of extraction efficiency, structural characterization, and performance optimization, which limits their potential industrial application [11]. Lignocellulosic fibers are hierarchical composite materials composed of cellulose microfibrils embedded in an amorphous matrix of hemicellulose, lignin, pectin, and surface waxes [12]. According to the literature, these fibers typically contain 40–70% cellulose, 10–30% hemicellulose, and 5–25% lignin, depending on plant species and extraction conditions [13]. These compositional variations strongly influence key properties such as tensile strength (200–1000 MPa), Young's modulus (5–70 GPa), and moisture absorption capacity, which may exceed 10% by weight under humid conditions [14]. In addition, structural parameters such as the crystallinity index (generally between 40% and 70%) and the microfibril angle (typically ranging from 5° to 20°) play a critical role in determining fiber stiffness and strength [15].

The presence of non-cellulosic components, particularly lignin and hemicellulose, constitutes a major limitation for fiber utilization. These amorphous constituents act as binding agents within the plant matrix, increasing hydrophilicity, reducing interfacial adhesion with polymer matrices, and limiting mechanical performance [16]. Therefore, their partial removal through appropriate extraction processes is essential to enhance fiber quality and functionality [17].

Fiber extraction is a crucial step that directly influences fiber morphology, chemical composition, and performance [18]. Traditional biological methods, such as water retting, are environmentally friendly but require long processing times, typically ranging from 7 to 21 days, and often result in inconsistent fiber quality due to uncontrolled microbial activity [19]. Chemical treatments, particularly alkaline extraction using sodium hydroxide (NaOH), are widely employed to remove hemicellulose and lignin and to improve fiber surface morphology and reactivity [20]. However, it has been reported that NaOH concentrations exceeding 5 wt% may lead to cellulose degradation, reduced degree of polymerization, and deterioration of mechanical properties [21].

To overcome these limitations, combined extraction methods integrating biological and chemical processes have been proposed as effective alternatives [22]. These hybrid approaches aim to balance efficiency and sustainability by enhancing the removal of non-cellulosic components while preserving cellulose integrity [23]. The effectiveness of combined treatments depends on several process parameters, including treatment duration, temperature, chemical concentration, and treatment sequence [24].

Recent studies have demonstrated that optimized combined extraction processes can increase cellulose content by up to 10–20%, significantly reduce lignin content, and improve fiber mechanical properties and surface morphology [25]. Furthermore, these treatments enhance fiber–matrix interfacial adhesion and contribute to improved composite performance [26]. Similar trends have been observed in various natural fibers subjected to hybrid treatments, confirming the relevance of combined approaches for advanced applications [27].

Despite the increasing interest in lignocellulosic fibers for sustainable composites, studies specifically dedicated to okra stem fibers remain limited, particularly regarding the comparative evaluation of biological, chemical, and combined extraction processes [28]. In addition, little information is available concerning the optimization of extraction parameters and their influence on the structural, physicochemical, and mechanical properties of okra fibers originating from Central Africa. Therefore, a systematic investigation is required to identify the most suitable extraction conditions for high-performance applications.

Another important aspect in fiber characterization is the analysis of the initial structural organization of plant tissues prior to extraction. Optical microscopy offers a simple, rapid, and effective technique for observing fiber bundle distribution and structural arrangement within lignocellulosic materials [29]. This technique allows a detailed analysis of morphological features, including fiber orientation, inter-fiber bonding zones, and structural heterogeneity within the lignocellulosic matrix, which are key factors governing fiber separation mechanisms and the efficiency of extraction processes [30].

In this study, optical microscopy was used to observe the bark of okra stems prior to any extraction process, providing insight into the initial organization of fiber bundles within the lignocellulosic matrix.

Therefore, the present work aims to compare biological, chemical, and combined extraction methods of okra fibers (*Abelmoschus esculentus*), while specifically optimizing the combined extraction process, based on physicochemical characterization and initial structural analysis.

## 2. Materials and Methods

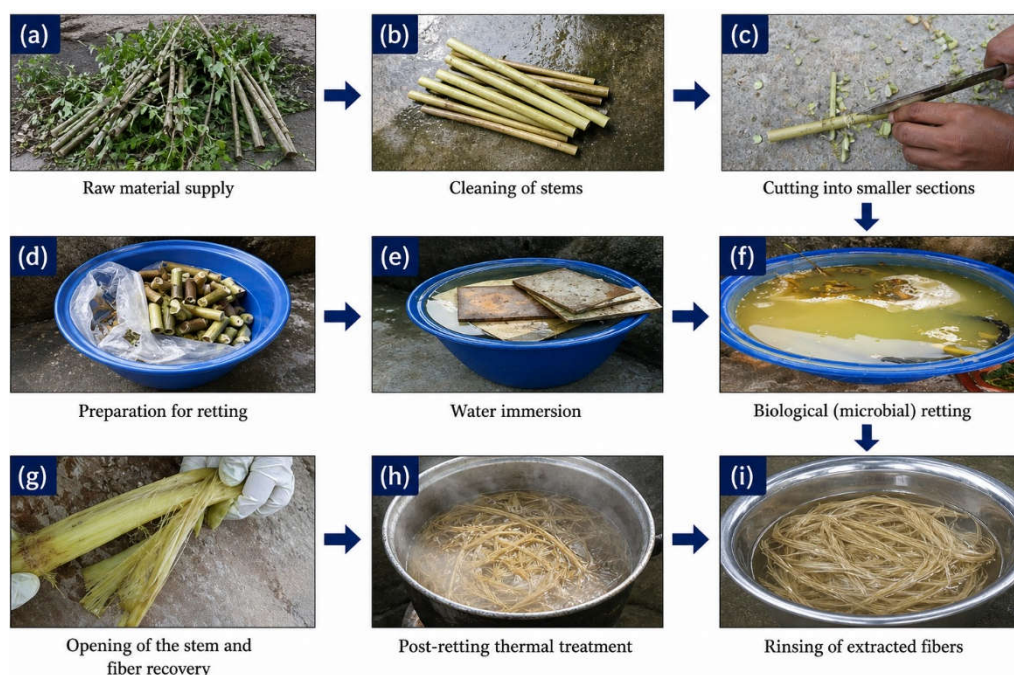
### 2.1. Raw Materials

Okra (*Abelmoschus esculentus*) stems were collected from local agricultural fields in the Littoral Region of Cameroon after harvesting. The bark was manually separated from the stems, and sections of  $50 \pm 5$  mm in length were cut using a hand saw and subsequently washed with water to remove impurities. Sodium hydroxide (NaOH, 99% purity) and acetic acid were supplied by Laboratory PYCNOLAB (Douala, Cameroon). Water used for retting was supplied by CAMWATER. Fibers were extracted by water retting (Water Ext) and chemically using NaOH solutions at concentrations of 2.5%, 5%, and 7.5%, selected based on previous studies reported by Soppie et al. [18] and Obame et al. [25]. For the combined extraction process, fibers obtained after retting were subsequently subjected to alkaline treatment under the same conditions in order to evaluate the influence of processing parameters on fiber properties.

### 2.2. Biological Extraction (Extraction in Stagnant Water)

Fiber extraction was carried out according to the following procedure. Okra (*Abelmoschus esculentus*) stems were cut into sections of approximately **85 cm** in length to ensure complete immersion in a tank containing **200 L of water** at ambient temperature. The stems were placed in bags and fully submerged to initiate the retting process, a widely used technique for lignocellulosic fiber extraction due to its efficiency and relatively low environmental impact [14]. After **14 days**, fibers were extracted from a total of **150 stems**, following the progressive microbial degradation of plant tissues, which facilitates the separation of fiber bundles [15]. This biological degradation process contributes to the partial removal of amorphous components such as hemicellulose and pectins, thereby improving fiber separation efficiency [14]. The extracted fibers were subsequently subjected to a thermal treatment consisting of boiling for **15 minutes**, aimed at enhancing fiber cleanliness and removing residual non-cellulosic materials [18]. Such post-retting treatments are commonly employed to improve fiber quality and surface characteristics [20]. After treatment, the fibers were thoroughly washed with running water to eliminate residual impurities and then air-dried for **24 hours** to reduce moisture content. This step is essential for stabilizing the physicochemical properties of the fibers and preventing degradation [21].

Finally, the dried fibers were stored in plastic bags prior to characterization. Appropriate storage conditions are necessary to preserve fiber integrity and minimize the influence of environmental humidity on their properties [26]. **Figure 1** presents a summary of the different stages of biological extraction.



**Figure 1.** Process of natural fiber extraction by biological retting: (a) Raw material supply; (b) Cleaning of stems; (c) Cutting into smaller sections; (d) Preparation for retting; (e) Water immersion; (f) Biological (microbial) retting; (g) Opening of the stem and fiber recovery; (h) post-retting thermal treatment; (i) Rinsing of extracted fibers.

### 2.3. Chemical Extraction in Sodium Hydroxide (NaOH)

Chemical treatment of the fibers was carried out according to the following procedure. Three samples of okra stems, each weighing 200 g, were measured using a digital balance and placed in plastic containers. Sodium hydroxide (NaOH) masses of 40 g, 80 g, and 120 g were accurately weighed and dissolved in 1600 mL of water, followed by stirring until a homogeneous solution was obtained. These concentrations correspond to 2.5 wt%, 5 wt%, and 7.5 wt%, which are commonly used for alkaline treatment of lignocellulosic fibers to remove amorphous components such as hemicellulose and lignin [12,13]. The solution was heated to boiling using a hot plate, after which 200 g of okra stems were introduced into the alkaline bath. After 15 minutes, the fibers were removed and extracted. Alkaline treatment is known to improve fiber cleanliness, increase surface roughness, and enhance the removal of non-cellulosic materials [18]. The extracted fibers were washed with 500 mL of distilled water to remove residual alkaline substances, then immersed in 300 mL of 0.1% acetic acid solution for 1 hour to neutralize the NaOH. This neutralization step is essential to stabilize the chemical structure of the fibers and prevent further degradation [25]. After neutralization, the fibers were rinsed again with 500 mL of distilled water, then placed in sieves for draining over 12 hours. Finally, the fibers were oven-dried at 60 °C for 24 hours to reduce moisture content and stabilize their physicochemical properties prior to characterization [21].

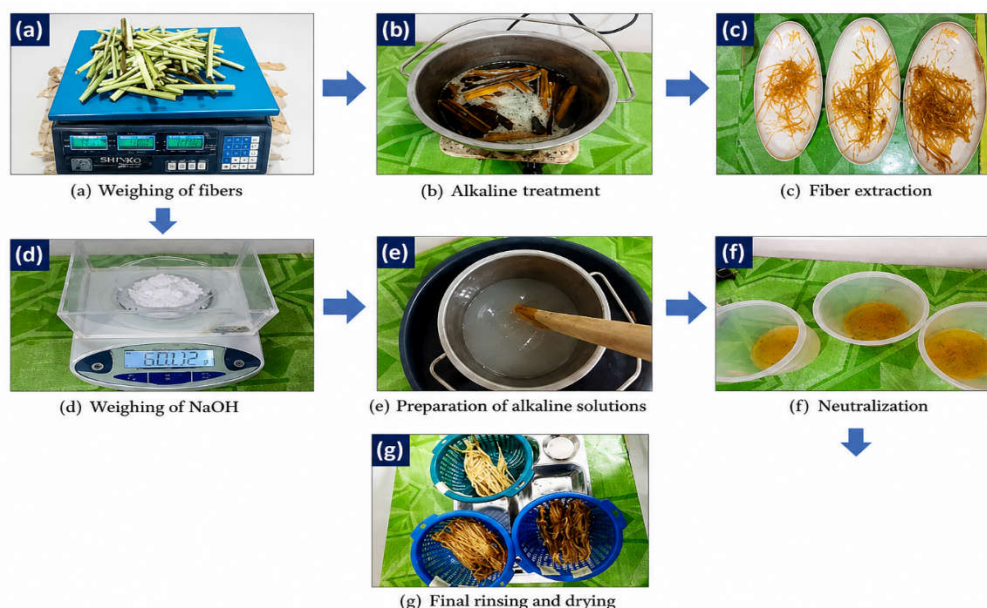
To evaluate the efficiency of the extraction process, the extraction yield ( $Rdt$ ) was determined by measuring the initial mass of the raw material ( $M_e$ ) and the dry mass of the extracted fibers after drying ( $M_s$ ) using a digital balance. **Figure 2** presents a summary of the different stages of chemical extraction.

The extraction yield was calculated according to Equation (1) [7]:

$$Rdt(\%) = \frac{M_s}{M_e} \times 100 \quad (1)$$

where:

- $M_e$  is the initial mass of the raw material (g),
- $M_s$  is the dry mass of the extracted fibers (g).



**Figure 2.** Chemical extraction process of natural fibers: (a) Weighing of raw fibers; (b) Alkaline treatment; (c) Fiber extraction after treatment; (d) Weighing of NaOH; (e) Preparation of alkaline solution; (f) Neutralization of treated fibers; (g) Final rinsing and drying of extracted fibers.

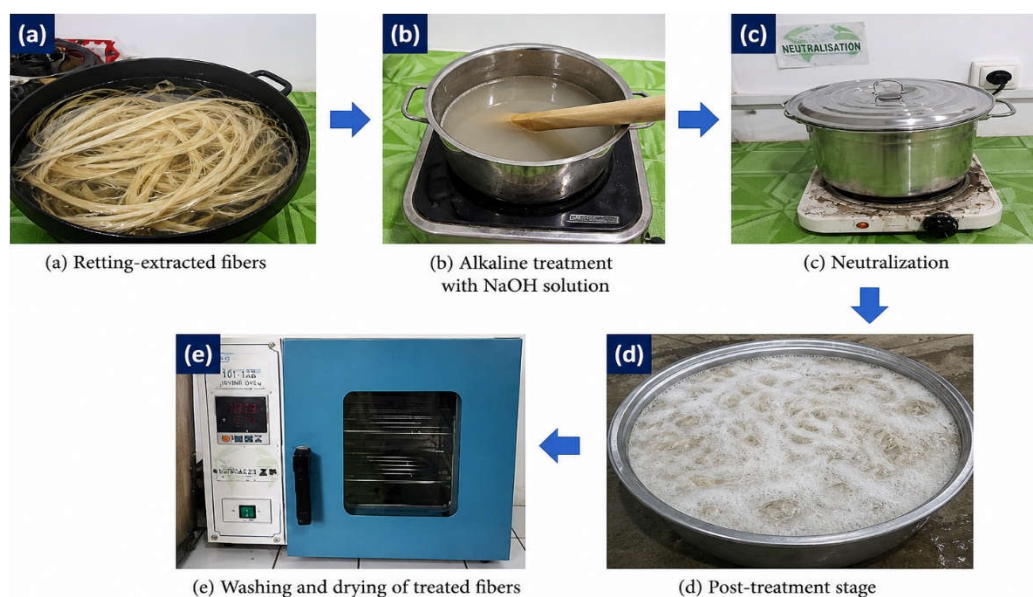
#### 2.4. Combined Extraction (Water Retting and Chemical Treatment)

The combined extraction process was carried out through a sequential treatment involving water retting followed by alkaline chemical treatment. According to each formulation, alkaline solutions were prepared in a water bath and heated to the desired temperature. The fibers obtained after retting were then introduced into the solution, stirred, and maintained under controlled conditions for a specified treatment time. At the end of the treatment, heating was stopped, and the fibers were separated using a sieve. The fibers were subsequently washed with running water and then rinsed with distilled water to remove residual chemicals. This alkaline treatment is known to enhance the removal of non-cellulosic components such as hemicellulose and lignin, thereby improving fiber surface properties and cleanliness [12,13,18]. The fibers were then immersed in 350 mL of acetic acid solution to neutralize the residual alkalinity of NaOH. This neutralization step is essential to stabilize the chemical structure of the fibers and prevent further degradation [25]. After neutralization, it was observed that the fibers remained agglomerated. To facilitate their separation and alignment, the fibers were treated in a hydrogen peroxide ( $H_2O_2$ ) bath for 1.5 h at a temperature between 85 and 90 °C. This oxidative treatment contributes to fiber individualization and improves fiber morphology by removing residual impurities.

Finally, the fibers were rinsed with water, air-dried, and subsequently oven-dried at 60 °C for 24 hours to reduce moisture content and stabilize their physicochemical properties before storage [21]. **Figure 3** presents a summary of the different stages of combined extraction.

#### 2.5. Optical Microscopy Analysis

The morphological analysis of *Abelmoschus esculentus* fibers was performed using an optical microscope. Observations were carried out on stem bark samples prior to extraction, focusing on both external and internal layers. Samples were prepared without specific treatment and observed at different magnifications to examine fiber structural organization. This technique is commonly used to investigate the morphology and heterogeneity of natural fibers [30].



**Figure 3.** post-treatment of retted natural fibers by alkaline process: (a) Retting-extracted fibers; (b) Alkaline treatment with NaOH solution; (c) Neutralization; (d) post-treatment stage; (e) Washing and drying of treated fibers.

## 2.6. Physical Characterization

### 2.6.1. Determination of Fiber Linear Density (Fineness)

The linear density (Tex), defined as the mass per unit length, was determined to evaluate the fineness of *Abelmoschus esculentus* fibers, a key parameter influencing their physical properties and textile performance. Fibers were cut to a known length ( $L$ ) and weighed ( $M$ ) using a precision balance. The linear density was calculated using Equation (2):

$$Tex = \frac{M}{L} \times 1000 \quad (2)$$

where:  $M$  is the mass (g) and  $L$  is the length (m). Lower Tex values indicate finer fibers, while higher values correspond to coarser fibers. This parameter is commonly used in the characterization of natural fibers [26,29].

### 2.6.2. Determination of Absolute Density of *Abelmoschus Esculentus* Fibers

The absolute density of the fibers was determined according to ISO 1973 (2021 edition) using a pycnometer method with ethanol as the reference liquid. This parameter is essential for characterizing natural fibers and assessing their suitability for textile and composite applications [26,29]. The absolute density ( $\rho_{abs}$ ) was calculated using Equation (3):

$$\rho_{abs} = \frac{M_0 \cdot \rho_{eth}}{M_1 - M_2} \quad (3)$$

where :

- $M_0$ (g) is the mass of the dry sample,
- $M_1$ (g) is the mass of the pycnometer filled with ethanol,
- $M_2$ (g) is the mass of the pycnometer containing ethanol and the sample,
- $\rho_{eth}$  is the density of ethanol (0.789 g/cm<sup>3</sup>).

Prior to measurements, samples were dried to eliminate moisture. This method provides a reliable estimation of the true fiber density, taking into account their internal structure and porosity [26].

### 2.6.3. Moisture Regain of Abelmoschus Esculentus Fibers

The moisture regain of the fibers was determined according to ISO 483:2005, using saturated saline solutions to control the relative humidity. After drying, the samples were conditioned in a desiccator for 24 hours. The Moisture regain ( $RH$ ) was calculated using Equation (4):

$$RH(\%) = \frac{M_f - M_i}{M_i} \times 100 \quad (4)$$

where :

- $M_i$ (g) is the initial dry mass of the sample,
- $M_f$ (g) is the mass after conditioning,
- $RH$ (%) represents the moisture regain after 24 h.

This parameter is essential for evaluating the hygroscopic behavior of natural fibers, which is strongly influenced by hydrophilic constituents such as cellulose and hemicellulose [14,26].

### 2.6.4. Water Absorption of Abelmoschus Esculentus Fibers

Water absorption of the fibers was determined according to NF EN ISO 1097-6, using the pycnometer method. This property reflects the ability of fibers to absorb water and is closely related to their porous structure and chemical composition.

After initial drying, the samples were immersed in water until saturation. The mass of the saturated surface-dry fibers was then measured. The water absorption ( $AB$ ) was calculated using Equation (5):

$$AB(\%) = \frac{M_{sss} - M_0}{M_0} \times 100 \quad (5)$$

where :

- $M_0$ (g) is the initial dry mass of the sample,
- $M_{sss}$ (g) is the saturated surface-dry mass,
- $AB$ (%) is the water absorption.

This parameter is strongly influenced by the presence of hydroxyl groups in cellulose and hemicellulose, which are responsible for the hydrophilic behavior of lignocellulosic fibers [14,26]. Higher water absorption generally indicates higher porosity and amorphous content within the fiber structure [14].

### 2.6.5. Mechanical Characterization of Abelmoschus Esculentus Fibers

Tensile tests were carried out in accordance with the protocol of standard NF T25-501-2 [1,12,21]. Fiber samples were prepared with a gauge length ( $L_0$ ) of **20 mm** and subjected to a maximum load of **5 kN** at a crosshead speed of **5 mm/min** at room temperature.

A total of **25 specimens** were tested for each extraction method, resulting in **100 samples** overall. This ensured the reproducibility and statistical reliability of the results.

**Figure 4** present a summary of tensile testing setup and specimen configuration for okra. The tensile strength at break ( $\sigma_r$ ) and the strain at break ( $\varepsilon_r$ ) were calculated using Equations (6) and (7) [6]:

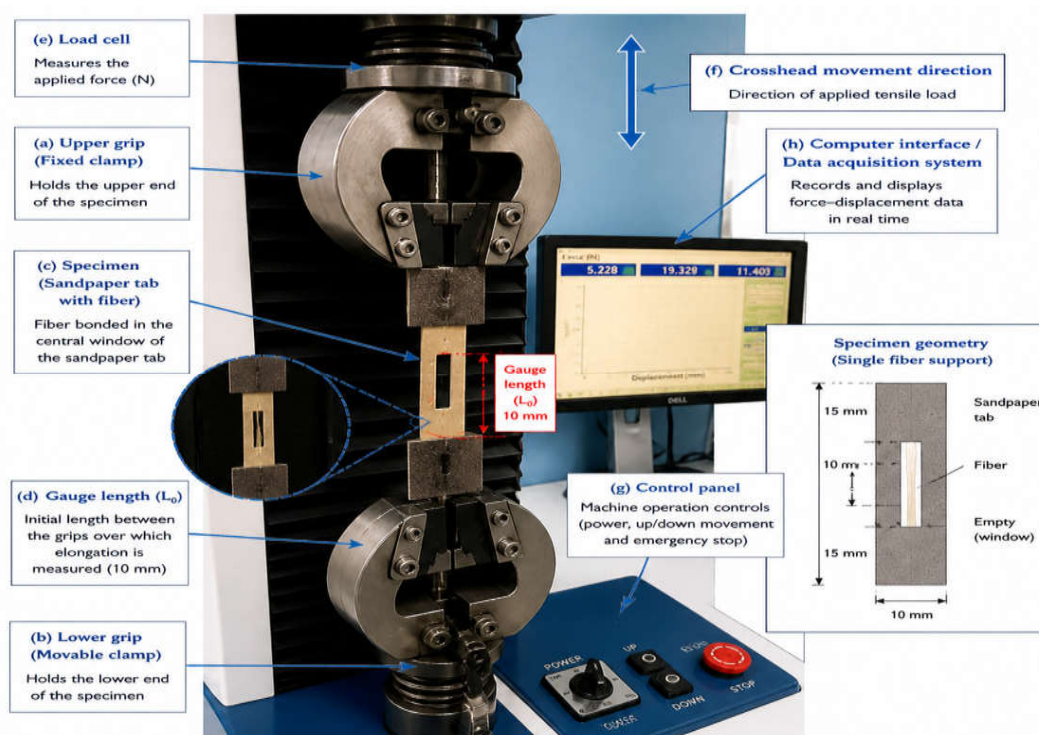
$$\sigma = \frac{F}{S} \quad (6)$$

$$\varepsilon = \frac{\Delta l}{L_0} \quad (7)$$

where:

- $F$  is the applied force (N),
- $S$  is the cross-sectional area of the fiber (mm<sup>2</sup>),
- $\sigma$  is the tensile stress (MPa),
- $\varepsilon$  is the strain,
- $\Delta l$  is the elongation (mm),
- $L_0$  is the initial gauge length (mm).

The Young's modulus ( $E$ ) was determined from the linear region of the stress–strain curve. These parameters are essential for evaluating the mechanical performance of natural fibers and their suitability for composite applications [19,27,28].



**Figure 4.** Tensile testing setup and specimen configuration for okra (*Abelmoschus esculentus*) fibers. (a) Upper grip (fixed clamp); (b) Lower grip (movable clamp); (c) Fiber specimen mounted on sandpaper tabs; (d) Gauge length ( $L_0 = 10$  mm); (e) Load cell; (f) Crosshead movement direction; (g) Control panel; (h) Computer interface/data acquisition system. The single fiber is fixed between sandpaper tabs to ensure proper alignment and avoid slippage during tensile loading.

### 2.7. Chemical Composition

The chemical composition of *Abelmoschus esculentus* fibers was determined to evaluate cellulose, hemicellulose, and lignin contents. The analyses were carried out using standard methods commonly applied to lignocellulosic materials. Prior to analysis, the fibers were dried to constant weight to remove residual moisture. The contents of cellulose, hemicellulose, and lignin were then determined using chemical methods based on the selective dissolution of lignocellulosic components. These methods enable the quantification of the main structural constituents of natural fibers and are widely used in biomaterial characterization [5,22,26].

### 2.8. Optimization of the Combined Extraction Process

The optimization of the combined extraction process (water retting followed by alkaline treatment) was carried out by investigating the influence of three operating parameters, namely temperature, sodium hydroxide (NaOH) concentration, and treatment time. These parameters are known to significantly affect the removal of amorphous components such as hemicellulose and lignin in natural fibers [12,13]. Alkaline treatment plays a key role in modifying fiber structure by improving surface cleanliness and properties, which justifies the selection of the experimental levels used in this study [18]. A factorial experimental design was implemented by combining two levels for each parameter:

- Temperature: 50 °C and 100 °C

- NaOH concentration: 1% and 3%
- Treatment time: 30 and 60 min

A total of eight (08) experimental runs were conducted to evaluate the individual and combined effects of these parameters. The performance of [12,13] the extracted fibers was evaluated through several physicochemical and mechanical properties. Physical properties such as water absorption, moisture regain, linear density, and density were considered as key indicators of fiber behavior [26]. Mechanical properties, including Young's modulus, tensile strength, and elongation at break, were also analyzed due to their importance in composite applications [27]. In addition, the chemical composition of the fibers (cellulose, hemicellulose, and lignin) was evaluated to assess the effect of extraction parameters on the lignocellulosic structure [25]. The optimization approach consisted of analyzing the influence of these parameters to determine the most favorable operating conditions for obtaining fibers with improved performance.

### 2.9. Fourier Transform Infrared (FTIR)

The functional groups present in *Abelmoschus esculentus* fibers were identified using Fourier Transform Infrared (FTIR) spectroscopy. The analyses were performed using a Shimadzu IRAffinity-1 CE spectrometer equipped with an ATR module and controlled by dedicated acquisition software. A few milligrams of finely ground fiber samples were analyzed over a spectral range of 4000–400  $\text{cm}^{-1}$ , with a resolution of 4  $\text{cm}^{-1}$  and 24 scans. The spectra were recorded in absorbance mode. This technique allows the identification of characteristic functional groups of lignocellulosic fibers, such as hydroxyl, carbonyl, and aromatic groups, and enables the assessment of chemical modifications induced by treatments [12,21,22].

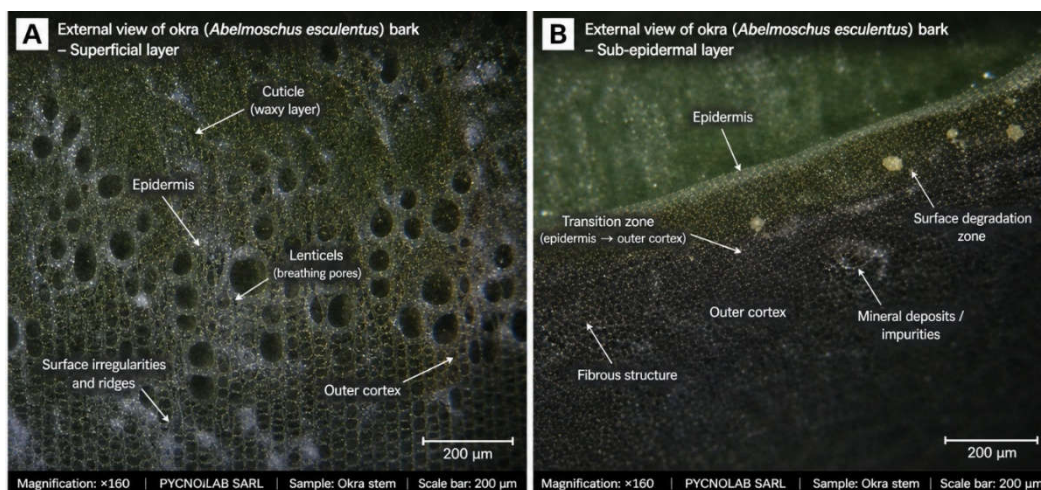
### 2.10. X-Ray Diffraction (XRD)

The crystalline structure of *Abelmoschus esculentus* fibers was analyzed using X-ray diffraction (XRD). The analyses were performed using an X-ray diffractometer with Cu-K $\alpha$  radiation ( $\lambda \approx 1.54 \text{ \AA}$ ) under standard operating conditions. The fiber samples were dried and finely ground prior to analysis. Measurements were carried out over a  $2\theta$  range of  $5^\circ$  to  $60^\circ$ , with an appropriate scanning rate. The obtained diffractograms were used to identify crystalline phases and evaluate the crystallinity of the fibers. This technique is widely used to characterize the crystalline structure of lignocellulosic fibers and to assess the effect of treatments on their molecular organization [22,28,29].

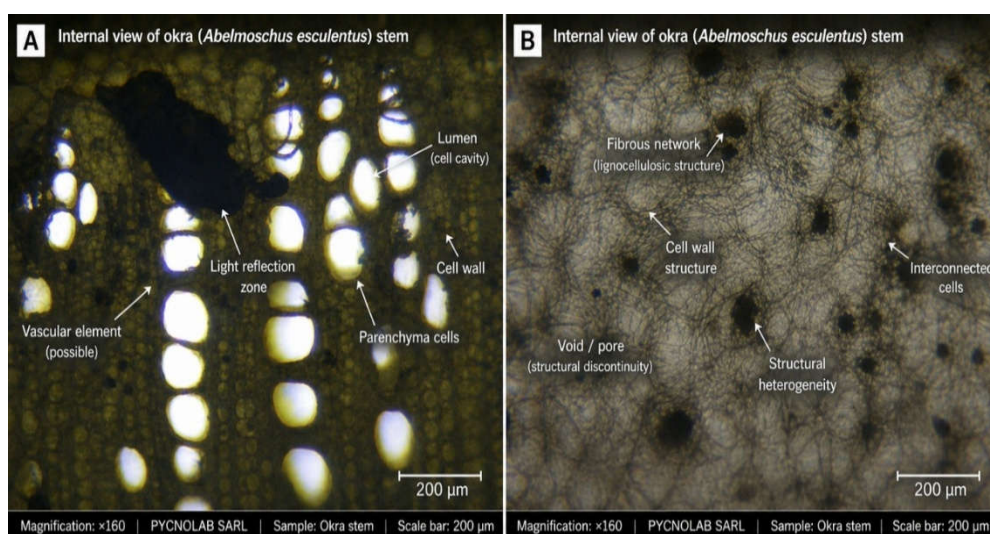
## 3. Results

### 3.1. Result of Optical Microscopy Analysis of *Abelmoschus Esculentus* Fibers

Optical micrographs of okra (*Abelmoschus esculentus*) stem bark reveal a heterogeneous and organized lignocellulosic structure, as shown in **Figures 5 and 6**. The external surface (**Figure 5A**) shows an epidermis covered by a cuticular layer with irregular features and pore-like structures, possibly corresponding to lenticels or degraded regions [31]. Beneath this layer, the sub-epidermal region (**Figure 5B**) exhibits a denser fibrous organization associated with cellulose, hemicellulose, and lignin [32]. The presence of particulate deposits indicates residual impurities from extraction processes [33], while localized degradation suggests structural alteration during processing [34]. The internal structure (**Figure 6**) exhibits large lumen-like cavities (**Figure 6A**) corresponding to vascular or parenchymal elements, contributing to the intrinsic porosity of the material [35]. In addition, a dense and interconnected fibrous network is observed (**Figure 6B**), characteristic of lignocellulosic materials and responsible for mechanical strength [32]. Structural heterogeneity and voids are also present, likely resulting from natural anatomical variability [35] or extraction-induced modifications [33]. Overall, this hierarchical organization confirms the suitability of okra fibers for textile and composite applications [31]. However, the presence of impurities and structural irregularities highlights the need for optimized extraction and treatment processes to improve fiber quality and performance [33].



**Figure 5.** Optical microscopy images of okra (*Abelmoschus esculentus*) stem bark highlighting its external organization. (A) The outermost layer exhibits an epidermal structure coated with a cuticular film and presenting pore-like features, which may correspond to lenticels or structural discontinuities induced during sample preparation. (B) The underlying region shows the transition toward the outer cortex, with a heterogeneous fibrous arrangement and the presence of particulate matter, suggesting surface contamination or mineral residues. Images were acquired using an optical microscope at  $\times 160$  magnification. Scale bars:  $200\ \mu\text{m}$ .

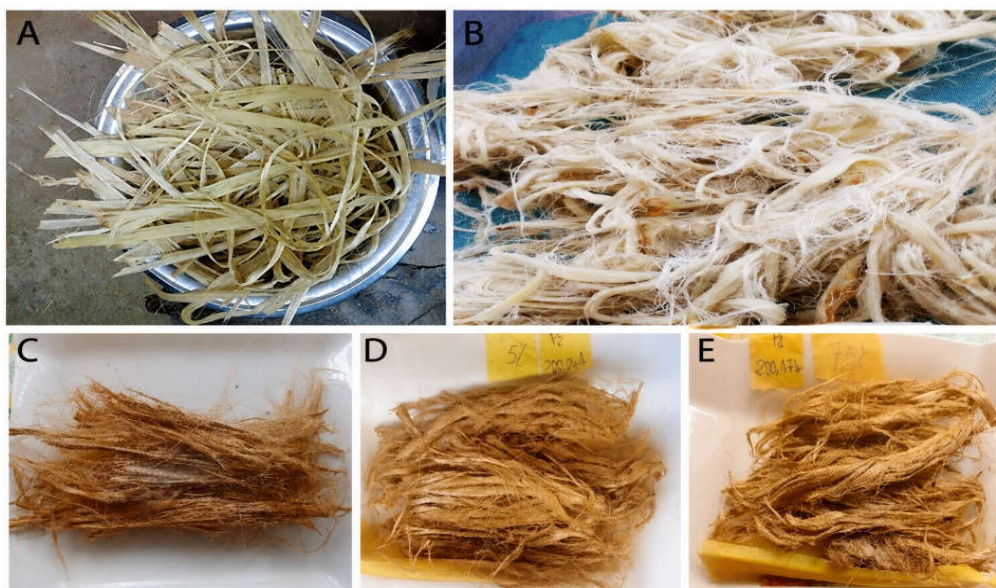


**Figure 6.** Optical micrographs of the internal structure of okra (*Abelmoschus esculentus*) stem. (A) Internal region showing large lumen-like structures corresponding to vascular elements or parenchymal cavities, with intense light reflection associated with cell wall boundaries and possible sample preparation effects. (B) Inner region revealing a dense fibrous network composed of interconnected cell walls, characteristic of lignocellulosic organization, with dark areas corresponding to voids or structural discontinuities.

### 3.2. Result of Extraction of Okra (*Abelmoschus Esculentus*) Fibers

The morphology of okra (*Abelmoschus esculentus*) fibers varies significantly with the extraction method (**Figure 7**). Biological extraction (**Figure 7A**) leads to partial fiber separation with remaining non-cellulosic materials [34]. The combined method (**Figure 7B**) improves fiber individualization and cleanliness, indicating more effective removal of lignin and hemicellulose [32]. Chemical treatment at 2.5% (**Figure 7C**) shows limited extraction efficiency, with compact bundles and impurities [33]. Increasing to 5% (**Figure 7D**) enhances fiber separation [32]. At 7.5% (**Figure 7E**), fibers are highly individualized but may exhibit structural weakening due to over-treatment [34]. Overall, chemical

concentration improves fiber quality, while the combined method provides a better balance between efficiency and structural preservation [35].



**Figure 7.** Morphological comparison of okra (*Abelmoschus esculentus*) fibers obtained using different extraction methods. (A) Biological extraction showing partially separated fibers with relatively smooth surfaces and residual non-cellulosic materials. (B) enhanced fiber individualization and cleaner surface morphology. (C) Chemical extraction at 2.5% showing compact fiber bundles with visible impurities and limited fibrillation. (D) Chemical extraction at 5% showing improved fiber separation and reduced impurities. (E) Chemical extraction at 7.5% showing highly individualized fibers with significant fiber separation, indicating increased removal of non-cellulosic components but possible structural weakening.

### 3.3. Results of Optimization Using RSM (Multi-Response Approach)

#### 3.3.1. Effect of Treatment on Hemicellulose Content

The response surface plots (**Figure 8**) show that hemicellulose content decreases with increasing NaOH concentration, temperature, and extraction time. This behavior is attributed to the dissolution of amorphous hemicellulose components under alkaline conditions [12,13]. The combined effect of temperature and concentration enhances the removal efficiency by accelerating hydrolysis reactions [21]. A slight plateau at higher durations suggests a limitation in accessible hemicellulose removal [5]. These findings are consistent with previous studies reporting effective hemicellulose reduction during alkaline treatment of natural fibers [10]. A second-order polynomial model was developed to describe the effect of temperature (T), NaOH concentration (C), and extraction time (t) on hemicellulose content. The relationship between the response and the independent variables is given by Equation (8):

$$H = 3.463 + 0.05194T + 2.819C + 0.256t - 0.03478TC - 0.002792Tt - 0.07065Ct + 0.000640TCt \quad (8)$$

where H represents the hemicellulose content (%), T is the temperature (°C), C is the NaOH concentration (%), and t is the extraction time (min).

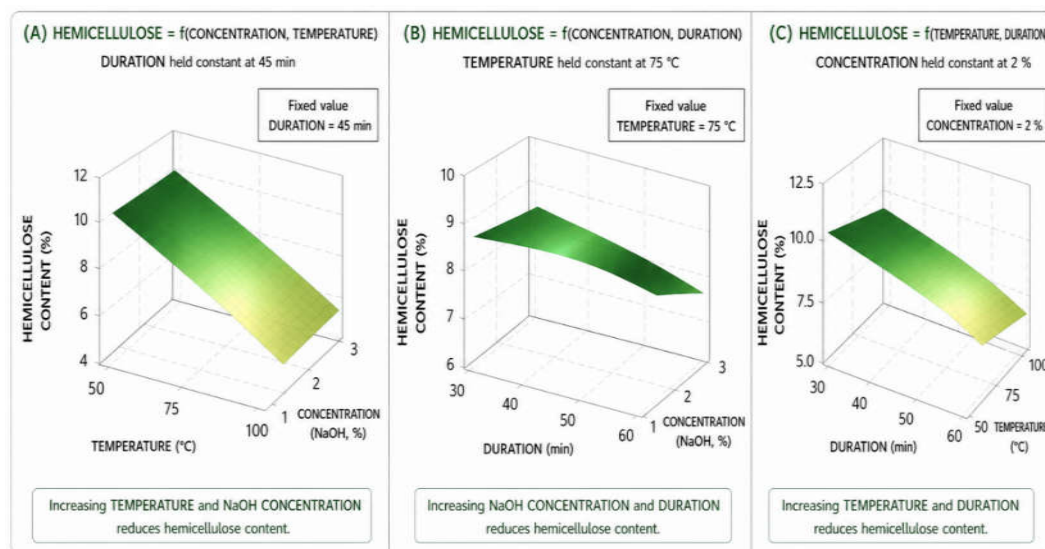
#### 3.3.2. Effect on Lignin

The response surface plots (**Figure 9**) indicate that lignin content decreases with increasing NaOH concentration, temperature, and extraction time. This reduction is attributed to the alkaline cleavage of lignin bonds and its solubilization [12,13]. The combined effect of temperature and concentration enhances delignification efficiency by accelerating reaction kinetics [21]. However, a plateau is observed at higher durations, suggesting limited accessible lignin removal [5]. These

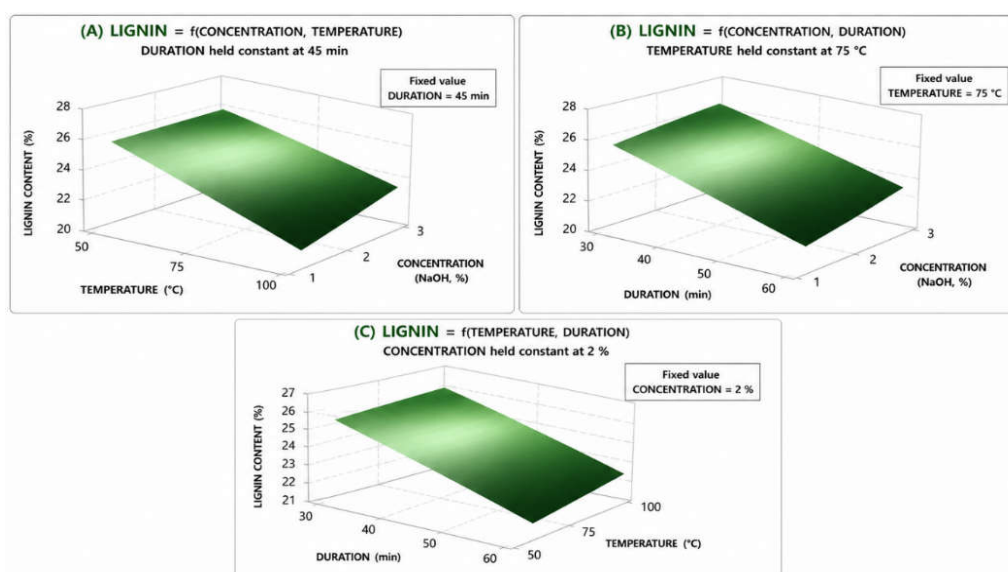
results are consistent with previous studies highlighting the effectiveness of alkaline treatment in improving fiber purity [33]. A regression model in uncoded units was developed to describe the effect of temperature (T), NaOH concentration (C), and extraction time (t) on lignin content. The relationship is expressed by Equation (9):

$$L = 77.07 - 0.5969T - 23.34C - 0.9215t + 0.2828TC + 0.009108Tt + 0.3773Ct - 0.004183Tct \quad (9)$$

where L represents the lignin content (%), T is the temperature (°C), C is the NaOH concentration (%), and t is the extraction time (min). The negative coefficients of temperature, concentration, and time indicate a decreasing trend in lignin content with increasing process severity.



**Figure 8.** Response surface plots showing the effect of NaOH concentration, temperature, and extraction time on hemicellulose content (%) of okra (*Abelmoschus esculentus*) fibers. (A) Effect of concentration and temperature at constant duration (45 min); (B) effect of concentration and duration at constant temperature (75 °C); and (C) effect of temperature and duration at constant NaOH concentration (2%). The results indicate a progressive reduction of hemicellulose content with increasing treatment severity.



**Figure 9.** Response surface plots showing the effect of NaOH concentration, temperature, and extraction time on lignin content (%) of okra (*Abelmoschus esculentus*) fibers. (A) Effect of concentration and temperature at constant duration (45 min); (B) effect of concentration and duration at constant temperature (75 °C); and (C)

effect of temperature and duration at constant NaOH concentration (2%). A significant decrease in lignin content is observed with increasing alkali concentration and temperature.

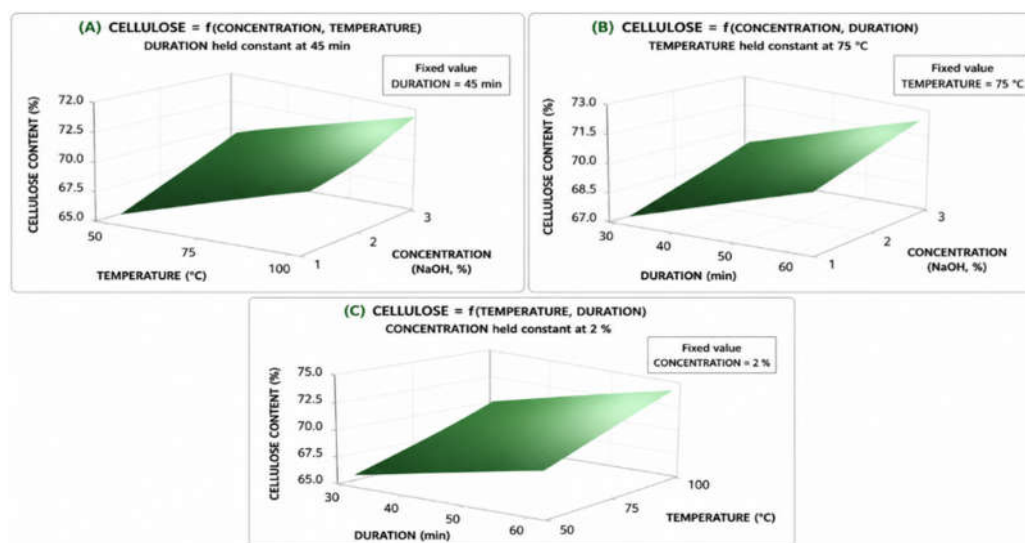
### 3.3.3. Effect on Cellulose

The response surface plots (**Figure 10**) indicate that cellulose content increases with increasing NaOH concentration, temperature, and extraction time. This trend is associated with the progressive removal of hemicellulose and lignin, leading to a relative enrichment of cellulose [12,13]. The combined effect of temperature and alkali enhances fiber purification by promoting the dissolution of amorphous components [21]. However, excessive treatment conditions may lead to partial cellulose degradation [5]. These results agree with previous studies reporting improved cellulose content after alkaline treatment of natural fibers [33].

A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on cellulose content. The relationship is expressed by Equation (10):

$$\text{Cellulose} = 19.41 + 0.5458T + 20.55C + 0.6664t - 0.2485TC - 0.006330Tt - 0.3073Ct + 0.003552Tt \quad (10)$$

where Cellulose represents the cellulose content (%),  $T$  is the temperature ( $^{\circ}\text{C}$ ),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min). The positive coefficients of temperature, concentration, and time indicate that cellulose content increases with increasing process severity. This behavior is attributed to the progressive removal of non-cellulosic components such as hemicellulose and lignin during alkaline treatment.



**Figure 10.** Response surface plots showing the effect of NaOH concentration, temperature, and extraction time on cellulose of okra (*Abelmoschus esculentus*) fibers. (A) Effect of concentration and temperature at constant duration (45 min); (B) effect of concentration and duration at constant temperature (75  $^{\circ}\text{C}$ ); and (C) effect of temperature and duration at constant NaOH concentration (2%). Cellulose content increases with increasing removal of non-cellulosic amorphous components.

### 3.3.4. Effect on Density

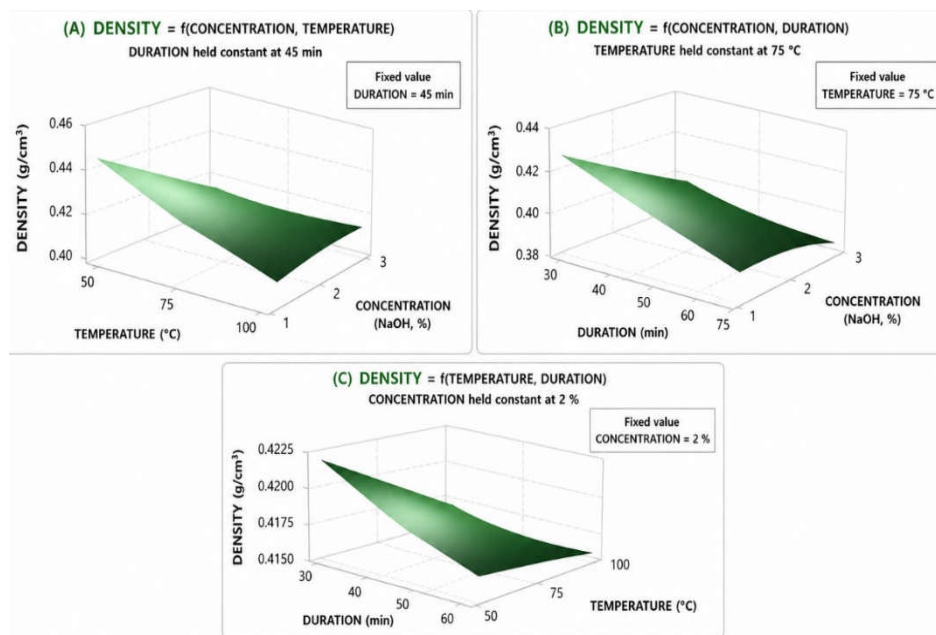
The response surface plots (**Figure 11**) indicate that fiber density increases with increasing treatment severity NaOH concentration, temperature, and extraction time. This behavior is associated with the progressive removal of lignin and hemicellulose, leading to a more porous fiber structure [12,13]. The combined effect of temperature and alkali enhances matrix dissolution, reducing compactness [21]. However, moderate conditions help preserve structural integrity and limit excessive density loss [5]. These findings are consistent with previous studies reporting

structural modification of natural fibers after alkaline treatment [33]. A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on density. The relationship is expressed by Equation (11):

$$\rho = 0.3365 + 0.001310T + 0.05150C + 0.004917t - 0.000790TC - 0.000060Tt - 0.002550Ct + 0.000031TCt \quad (11)$$

where  $\rho$  represents the density,  $T$  is the temperature ( $^{\circ}\text{C}$ ),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).

The positive coefficients of temperature, concentration, and time indicate an increase in density with increasing treatment severity. This trend may be attributed to the removal of amorphous components such as hemicellulose and lignin, leading to a more compact and cellulose-rich structure.



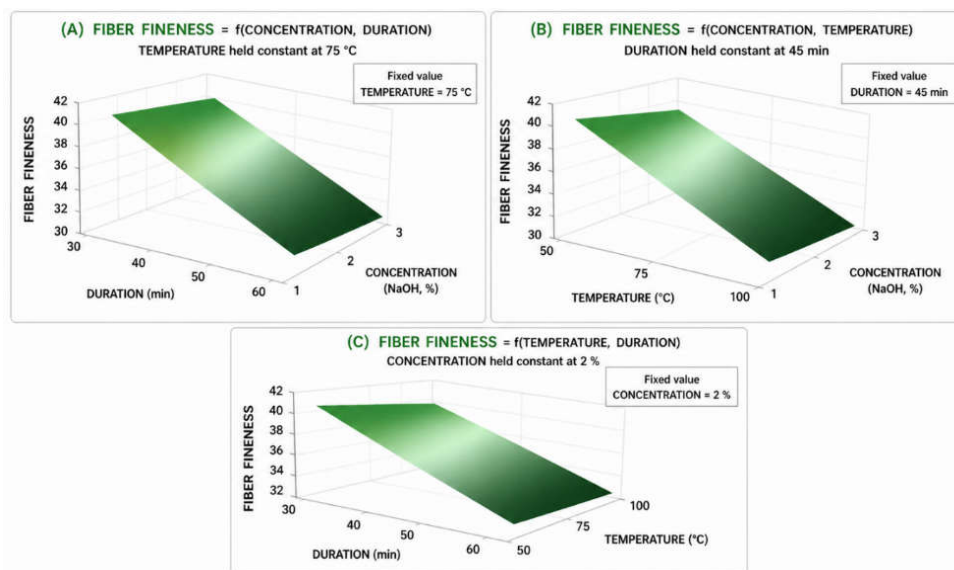
**Figure 11.** Response surface plots showing the effect of NaOH concentration, temperature, and extraction time on the density of okra (*Abelmoschus esculentus*) fibers. (A) Effect of concentration and temperature at constant duration (45 min); (B) effect of concentration and duration at constant temperature (75  $^{\circ}\text{C}$ ); and (C) effect of temperature and duration at constant NaOH concentration (2%). The results reflect structural modifications induced by alkaline treatment.

### 3.3.5. Effect on Fineness

The response surface plots (**Figure 12**) indicate that fiber fineness increases with increasing NaOH concentration, temperature, and extraction time. This behavior is attributed to the progressive removal of non-cellulosic components, particularly lignin and hemicellulose, leading to finer and more individualized fibers [12,13]. The combined effect of temperature and alkali enhances fiber separation by weakening inter-fibrillar bonding [21]. However, excessive treatment conditions may damage the cellulose structure and reduce fiber quality [5]. These results are consistent with previous studies reporting improved fiber fineness after alkaline treatment of natural fibers [33]. A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on fiber fineness. The relationship is expressed by Equation (12):

$$F = 67.10 - 0.5540T - 9.655C - 0.3270t + 0.2034TC + 0.005703Tt + 0.1223Ct - 0.002257TCt \quad (12)$$

where  $F$  represents the fiber fineness,  $T$  is the temperature ( $^{\circ}\text{C}$ ),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).



**Figure 12.** Fiber fineness (Tex) of *Abelmoschus esculentus* fibers obtained using biological, combined (EB1–EB8), and chemical extraction processes. The left panel shows variations among extraction conditions, while the right panel illustrates the effect of increasing NaOH concentration (2.5–7.5 wt%) on fiber fineness.

### 3.3.6. Effect on Water Absorption

The response surface plots (**Figure 13**) show that water absorption (WA%) increases with increasing NaOH concentration, temperature, and extraction time. This behavior is attributed to the removal of hemicellulose and lignin, which reduces the availability of hydrophilic groups in the fiber structure [12,13]. The combined effect of temperature and alkali enhances fiber surface modification, limiting moisture uptake [21]. However, moderate conditions maintain a balance between structural integrity and hydrophilicity [5]. These findings are consistent with previous studies reporting reduced water absorption after alkaline treatment of natural fibers [33]. A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on water absorption (WA). The relationship is expressed by Equation (13):

$$WA(\%) = 352.0 - 2.242T - 95.49C - 4.295t + 1.151TC + 0.04774Tt + 2.097Ct - 0.02428Tct \quad (13)$$

where WA represents the water absorption (%),  $T$  is the temperature (°C),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).

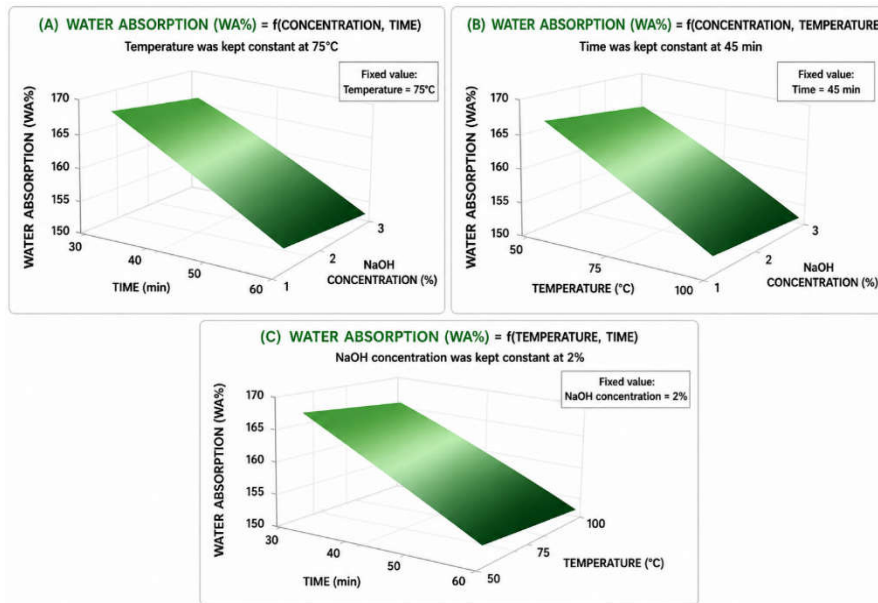
### 3.3.7. Effect on Moisture Regain

The response surface plots (**Figure 14**) indicate that moisture regain (MR%) decreases with increasing NaOH concentration, temperature, and extraction time. This reduction is attributed to the removal of hemicellulose and lignin, which are rich in hydrophilic groups responsible for moisture uptake [12,13]. The combined effect of temperature and alkali enhances fiber purification, thereby reducing water affinity [21]. However, moderate treatment conditions help preserve some moisture sorption capacity [5]. These findings are consistent with previous studies reporting reduced moisture regain after alkaline treatment of natural fibers [33].

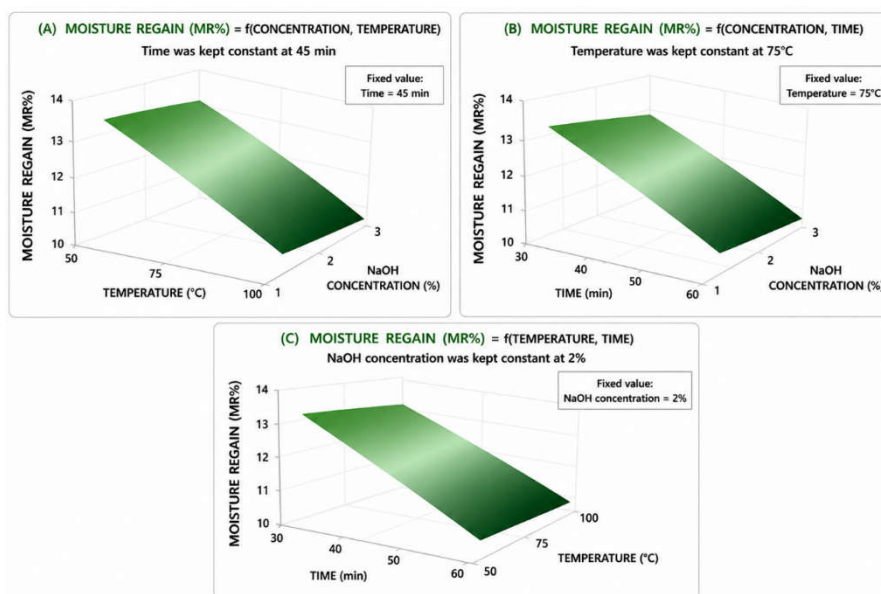
A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on moisture regain. The relationship is expressed by Equation (14):

$$R = 49.61 - 0.3989T - 12.62C - 0.6610t + 0.1289TC + 0.006917Tt + 0.2203Ct - 0.002363Tct \quad (14)$$

where R represents the moisture regain (%),  $T$  is the temperature (°C),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).



**Figure 13.** Water absorption capacity (%) of *Abelmoschus esculentus* fibers as a function of extraction method and NaOH concentration, showing variations related to the removal of non-cellulosic components.



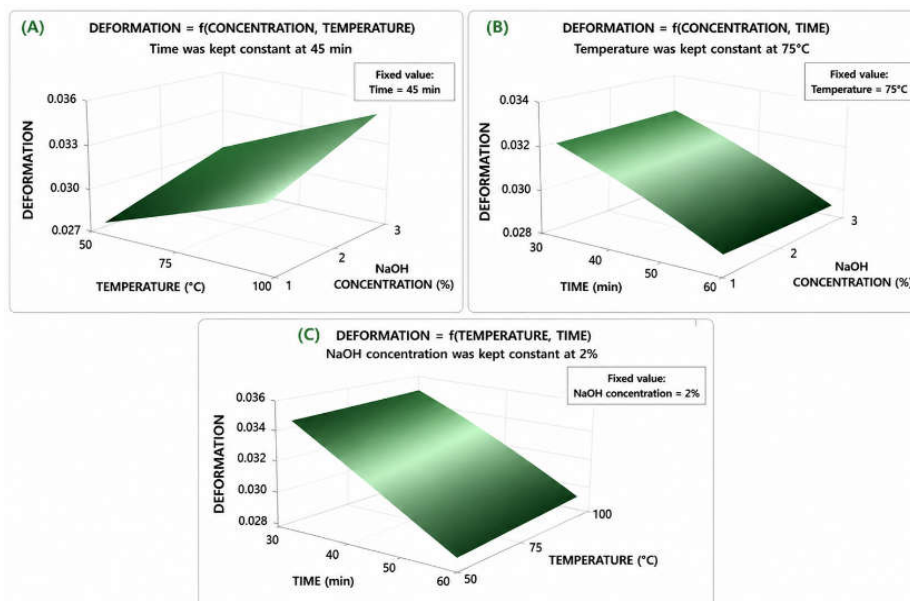
**Figure 14.** Moisture regain (%) of fibers extracted under different biological, combined, and chemical conditions. The results highlight the influence of NaOH concentration and processing parameters on the hygroscopic behavior of the fibers.

### 3.3.8. Deformation

The response surface plots (**Figure 15**) show that deformation increases with increasing NaOH concentration and temperature, while it decreases with prolonged extraction time. This behavior is associated with the progressive removal of lignin and hemicellulose, which enhances fiber flexibility and structural rearrangement [12,13]. The combined effect of temperature and alkali promotes fibrillation, leading to improved deformation capacity [21]. However, excessive treatment duration may weaken the cellulose network, reducing elasticity [5]. These findings are consistent with previous studies reporting structural modification of natural fibers under alkaline treatment [33]. A regression model in uncoded units was developed to describe the effect of temperature (T), NaOH concentration (C), and extraction time (t) on fiber deformation. The relationship is expressed by Equation (15):

$$D = -0.006400 + 0.000632T + 0.01090C + 0.000492t - 0.000202TC - 0.000010Tt - 0.000175Ct + 0.000004TCt \quad (15)$$

where  $D$  represents the deformation,  $T$  is the temperature ( $^{\circ}\text{C}$ ),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).



**Figure 15.** Response surface plots showing the effect of NaOH concentration, temperature, and extraction time on the deformation behavior of okra (*Abelmoschus esculentus*) fibers. (A) Effect of concentration and temperature at constant duration (45 min); (B) effect of concentration and duration at constant temperature (75  $^{\circ}\text{C}$ ); and (C) effect of temperature and duration at constant NaOH concentration (2%). The results indicate structural changes influencing fiber flexibility.

### 3.3.9. Tensile Strength

The response surface plots (**Figure 16**) indicate that tensile strength increases with NaOH concentration and moderate temperature, but slightly decreases at higher temperatures and prolonged treatment time. This trend is attributed to the removal of lignin and hemicellulose, which improves fiber alignment and load transfer efficiency [12,13]. The combined effect of alkali and temperature enhances fibrillation and interfacial bonding [21]. However, excessive treatment may damage the cellulose structure, leading to strength reduction [5]. These findings are consistent with previous studies reporting improved mechanical properties after controlled alkaline treatment of natural fibers [33]. A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on tensile stress. The relationship is expressed by Equation (16):

$$\sigma \text{ (MPa)} = 51.09 + 0.1971T + 12.90C + 0.3802t - 0.09910TC - 0.000170Tt + 0.2565Ct - 0.003430TCt \quad (16)$$

where  $\sigma$  represents the tensile stress (MPa),  $T$  is the temperature ( $^{\circ}\text{C}$ ),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).

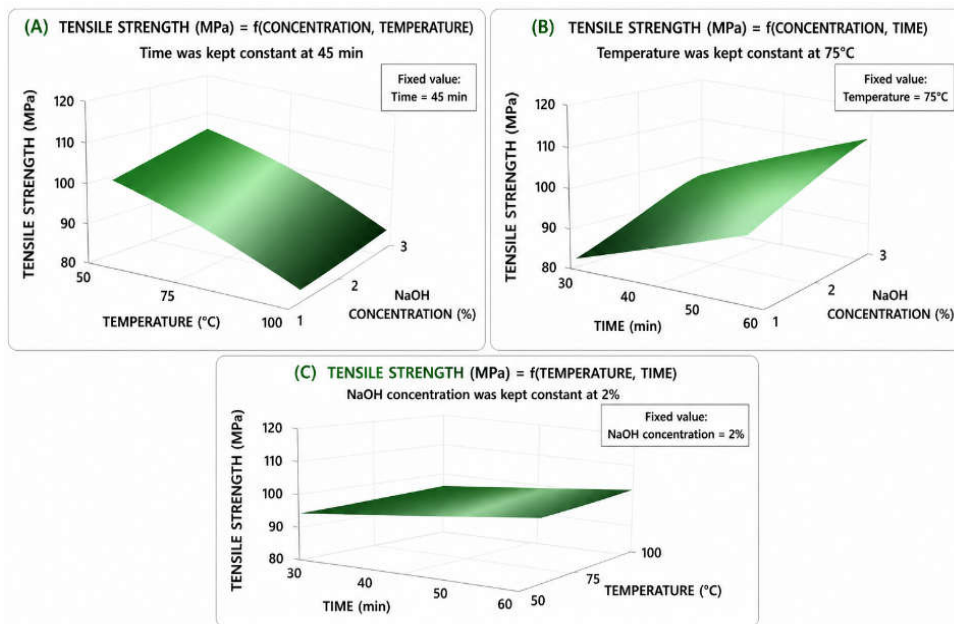
### 3.3.10. Young's Modulus

The response surface plots (**Figure 17**) indicate that Young's modulus increases with NaOH concentration and moderate temperature, but decreases slightly at higher temperatures and prolonged extraction time. This behavior is attributed to the removal of amorphous components such as lignin and hemicellulose, leading to increased cellulose crystallinity and stiffness [12,13]. The combined effect of alkali and temperature enhances fiber rigidity through structural reorganization [21]. However, excessive treatment may cause partial degradation of cellulose chains, reducing

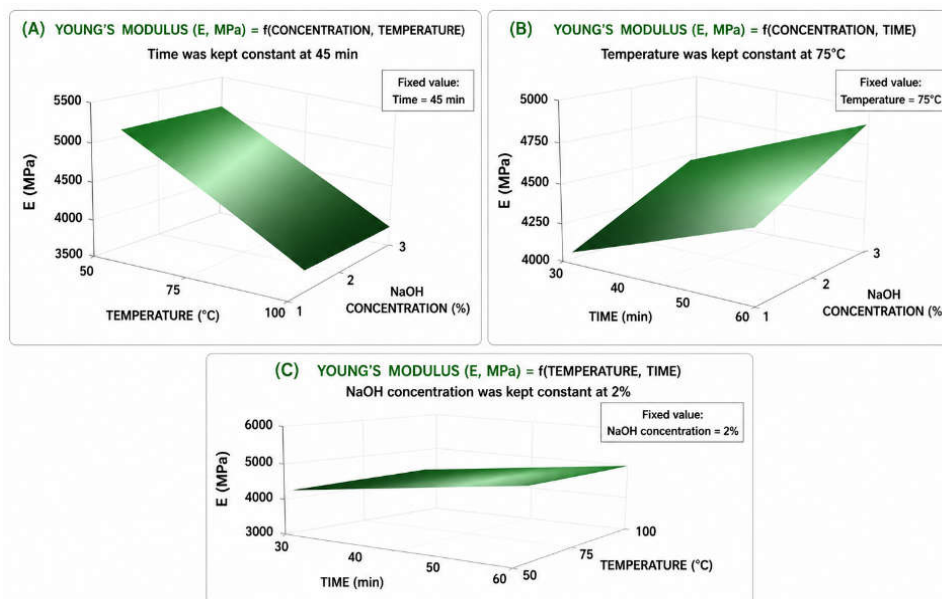
stiffness [5]. These findings are consistent with previous studies reporting improved mechanical performance after controlled alkaline treatment of natural fibers [33]. A regression model in uncoded units was developed to describe the effect of temperature ( $T$ ), NaOH concentration ( $C$ ), and extraction time ( $t$ ) on Young's modulus. The relationship is expressed by Equation (17):

$$E \text{ (MPa)} = 12114 - 124.4T - 3260C - 102.7t + 44.88TC + 1.823Tt + 65.78Ct - 0.9056Tct \quad (17)$$

where  $E$  represents the Young's modulus (MPa),  $T$  is the temperature ( $^{\circ}\text{C}$ ),  $C$  is the NaOH concentration (%), and  $t$  is the extraction time (min).



**Figure 16.** Tensile strength (MPa) of fibers extracted using biological, combined, and chemical methods, highlighting the effect of alkaline treatment on mechanical performance.



**Figure 17.** Response surface plots showing the effect of NaOH concentration, temperature, and extraction time on Young's modulus ( $E$ , MPa) of okra (*Abelmoschus esculentus*) fibers. (A) Effect of concentration and temperature at constant duration (45 min); (B) effect of concentration and duration at constant temperature (75

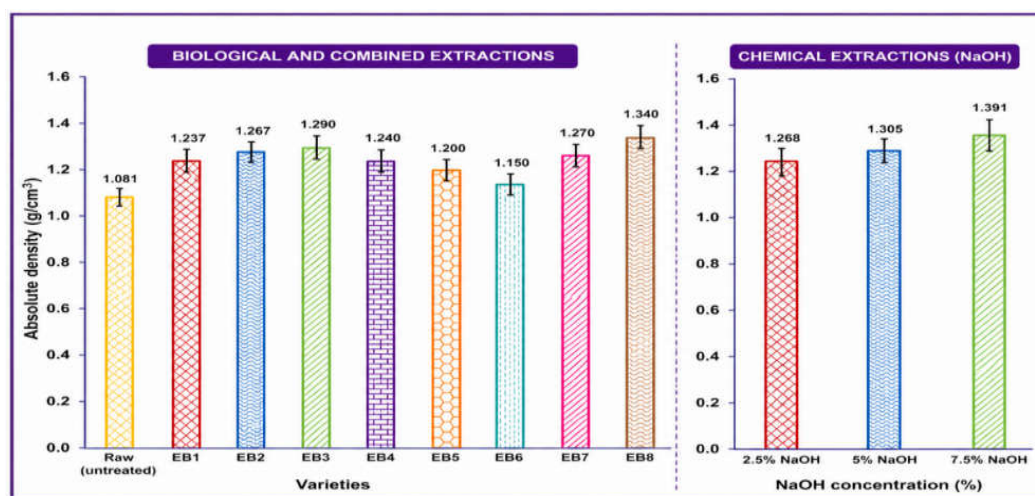
°C); and (C) effect of temperature and duration at constant NaOH concentration (2%). The results indicate increased stiffness associated with fiber purification and structural reorganization.

The statistical analysis of the experimental results was performed using ANOVA to evaluate the significance of the developed models. The results indicate that the models are statistically significant ( $p < 0.05$ ), confirming that the selected factors (temperature, NaOH concentration, and treatment time) have a significant influence on the studied responses. The coefficient of determination ( $R^2$ ) values obtained for the different responses were higher than 0.90, indicating a good agreement between experimental and predicted values. This demonstrates the reliability and accuracy of the developed regression models. Furthermore, the adjusted  $R^2$  values were in close agreement with the  $R^2$  values, confirming the adequacy of the models without overfitting. The lack-of-fit tests were found to be non-significant ( $p > 0.05$ ), indicating that the models are suitable for predicting the behavior of okra fibers under the studied conditions.

### 3.4. Results of Physical Characterization

#### 3.3.1. Linear Density (Fineness)

The absolute density of okra (*Abelmoschus esculentus*) fibers varies significantly with the extraction method and treatment conditions (**Figure 18**). For biological and combined extractions, the increase in density compared to raw fibers indicates partial removal of non-cellulosic components such as pectin and hemicellulose, leading to improved fiber compactness and organization [5]. This behavior is consistent with previous studies on natural fibers where mild treatments preserve structural integrity while enhancing fiber quality [10]. For chemical extraction, density increases with NaOH concentration (2.5% to 7.5%), confirming that alkaline treatment promotes delignification and removal of hemicellulose, resulting in denser fibers [12]. Similar trends have been reported for various lignocellulosic fibers, where chemical treatments improve fiber structure and interfacial properties [13]. At higher concentration (7.5%), the highest density is observed; however, excessive treatment may induce structural degradation despite improved cleanliness [24]. This phenomenon is widely reported in the literature, where over-treatment weakens fiber integrity [21]. Overall, both extraction methods enhance fiber density, but chemical treatment shows a stronger dependence on concentration. The combined method provides a better balance between efficiency and preservation of fiber structure, making it suitable for composite applications [11].

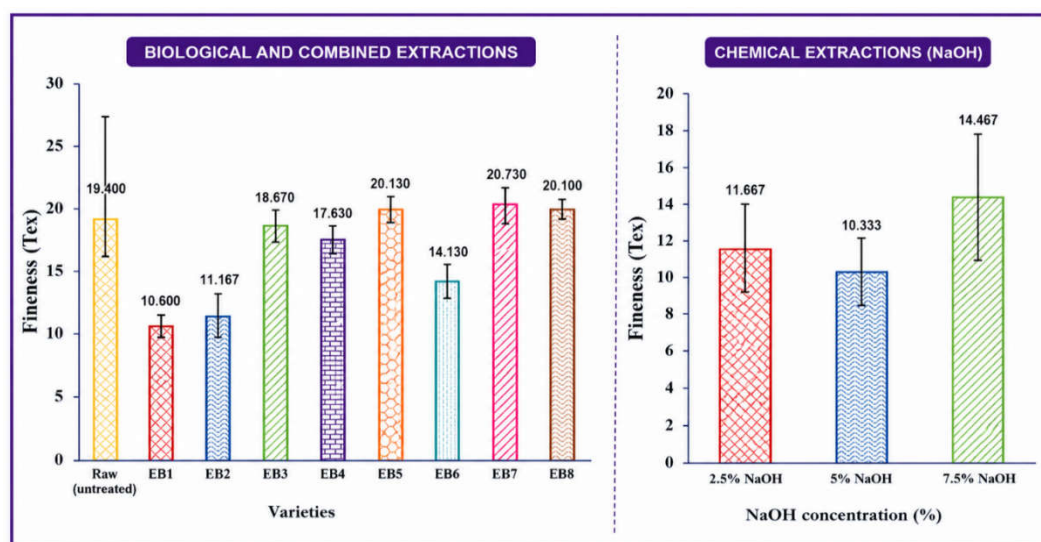


**Figure 18.** Absolute density of *Abelmoschus esculentus* fibers obtained from different extraction methods. The left panel shows fibers extracted by biological and combined processes (EB1–EB8), while the right panel presents fibers treated with different NaOH concentrations (2.5–7.5%). The results indicate variations in density depending on extraction conditions.

### 3.3.2. Fiber Fineness

The fineness of okra (*Abelmoschus esculentus*) fibers is strongly influenced by the extraction method and treatment conditions (**Figure 19**). For biological and combined extractions, a general decrease in fineness is observed compared to raw fibers, indicating progressive separation of fiber bundles into finer elementary fibers. This behavior is attributed to the partial removal of pectin and hemicellulose, which act as binding agents between fibers [5]. Similar trends have been reported for natural fibers where controlled extraction improves fiber individualization without severe structural damage [10]. For chemical extraction, fineness varies with NaOH concentration. At lower concentration (2.5%), fibers remain relatively coarse due to incomplete removal of non-cellulosic components [12]. At intermediate concentration (5%), a reduction in fineness indicates improved fiber separation [13]. At higher concentration (7.5%), an increase in fineness is observed, reflecting effective delignification and enhanced fibrillation [21]. However, excessive chemical treatment may also lead to fiber damage and irregular structure [24].

Overall, chemical treatment promotes better fiber individualization compared to biological methods, but requires optimization to avoid degradation. The combined extraction method offers a balanced approach, improving fineness while preserving fiber integrity, which is essential for textile and composite applications [11].

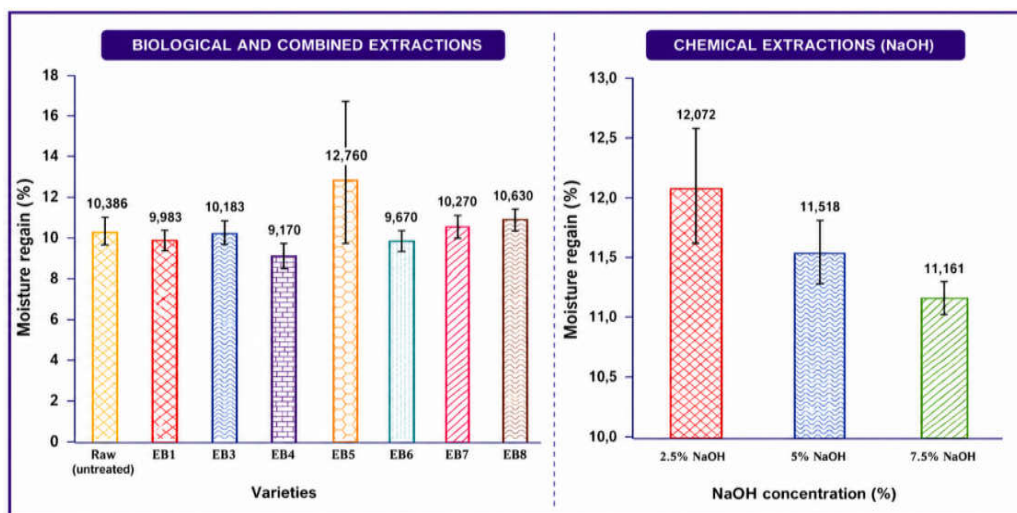


**Figure 19.** Fiber fineness (Tex) of *Abelmoschus esculentus* fibers obtained using biological, combined (EB1–EB8), and chemical extraction processes. The left panel shows variations among extraction conditions, while the right panel illustrates the effect of increasing NaOH concentration (2.5–7.5 wt%) on fiber fineness.

### 3.3.3. Moisture Regain

As shown in **Figure 20**, the moisture regain of okra (*Abelmoschus esculentus*) fibers is influenced by both extraction method and NaOH concentration. For biological and combined extractions, moisture regain values remain relatively stable, indicating that partial removal of non-cellulosic components preserves the hydrophilic nature of the fibers [5]. Variations among samples (EB1–EB8) are associated with differences in fiber structure and surface composition [10]. For chemical extraction, moisture regain decreases slightly with increasing NaOH concentration (2.5% to 7.5%). This trend is attributed to the removal of hemicellulose and other amorphous constituents responsible for water absorption [12]. Similar behavior has been reported for alkali-treated natural fibers, where treatment reduces hydroxyl group availability [13]. At higher concentration (7.5%), the lowest moisture regain is observed, indicating reduced hygroscopicity due to increased cellulose crystallinity [21]. However, excessive treatment may alter fiber structure and affect performance [24]. Overall, chemical treatment reduces moisture uptake, while biological methods maintain higher

hygroscopicity. This balance is critical depending on the intended application, particularly for composites where low moisture absorption is preferred [11].



**Figure 20.** Moisture regain of okra (*Abelmoschus esculentus*) fibers obtained by different extraction methods. The left panel shows biological and combined extractions (raw, EB1–EB8), while the right panel presents chemical extraction at different NaOH concentrations (2.5%, 5%, and 7.5%). Error bars represent standard deviation.

### 3.3.4. Water Absorption

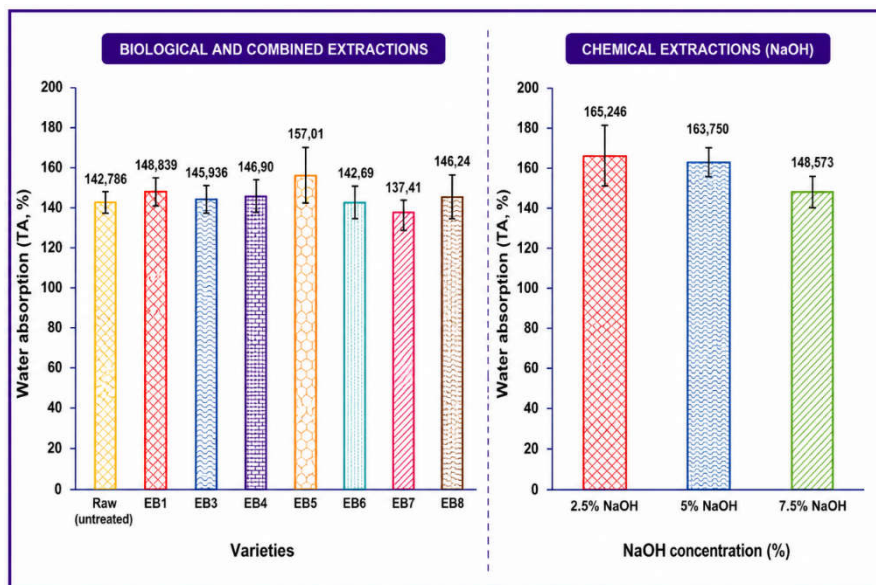
As shown in **Figure 21**, the water absorption behavior of okra (*Abelmoschus esculentus*) fibers is influenced by both extraction method and NaOH concentration. For **biological and combined extractions**, water absorption remains relatively high ( $\approx 137$ – $157\%$ ), which reflects the hydrophilic nature of lignocellulosic fibers rich in cellulose and hemicellulose [5]. Variations among samples (EB1–EB8) are related to differences in fiber structure, porosity, and residual non-cellulosic components [10]. For **chemical extraction**, water absorption decreases with increasing NaOH concentration (2.5% to 7.5%). This reduction is attributed to the removal of hemicellulose and amorphous components containing hydroxyl groups responsible for moisture uptake [12]. Similar behavior has been widely reported for alkali-treated natural fibers [13]. At higher concentration (7.5%), the lowest absorption is observed, indicating reduced hygroscopicity and improved fiber compactness due to increased cellulose crystallinity [21]. However, excessive treatment may lead to structural modification affecting fiber performance [24]. Overall, chemical treatment reduces water absorption, while biological methods preserve higher hydrophilicity. This balance is critical for applications such as composites, where low water uptake improves durability and interfacial stability [11].

## 3.5. Mechanical Characterization of *Abelmoschus Esculentus* Fibers

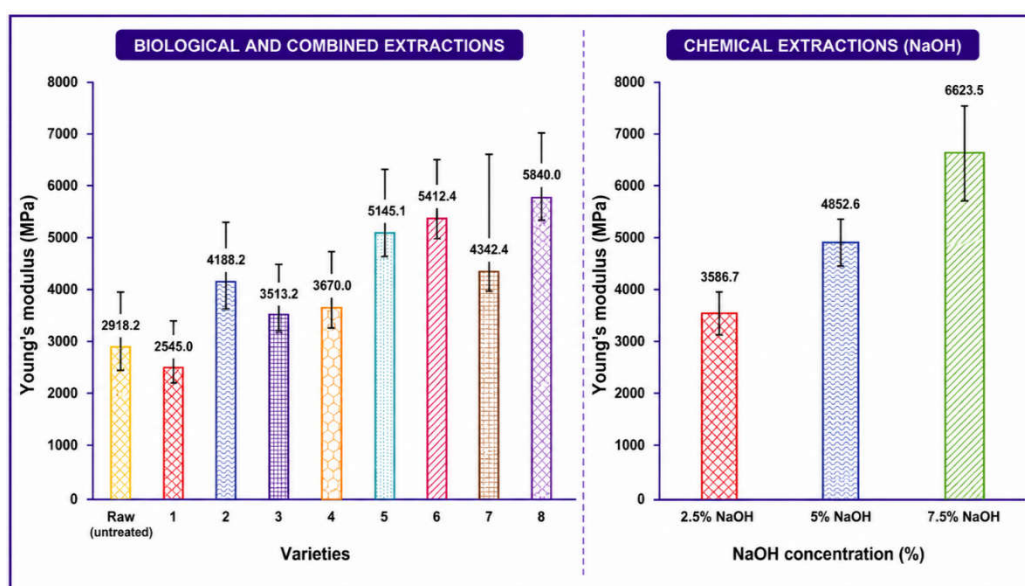
### 3.5.1. Young's Modulus

As shown in **Figure 22**, the Young's modulus of okra (*Abelmoschus esculentus*) fibers is strongly influenced by the extraction method and treatment conditions. For biological (retting) extraction, a progressive increase in Young's modulus is observed from raw fibers to treated samples (1–8). This trend is attributed to the gradual removal of pectins and hemicellulose, which enhances fiber individualization and improves load transfer within the cellulose microfibril structure [5]. Similar behavior has been reported for natural fibers subjected to retting processes, where partial degradation of binding components increases stiffness [17]. For chemical extraction, Young's modulus increases significantly with NaOH concentration (2.5% to 7.5%). This improvement is associated with the removal of lignin and hemicellulose, leading to higher cellulose crystallinity and better alignment of

microfibrils [12]. Such effects are widely reported in alkali-treated natural fibers, where chemical treatment enhances stiffness and mechanical performance [13,21]. At higher concentration (7.5%), the highest modulus is obtained, indicating effective purification and improved structural organization of cellulose. However, excessive alkali treatment may also induce partial degradation of cellulose chains, which can negatively affect mechanical properties beyond optimal conditions [24]. Overall, both extraction methods improve fiber stiffness, but chemical treatment shows a more pronounced effect due to efficient removal of non-cellulosic components. The combined optimization of extraction conditions is therefore essential to achieve high mechanical performance while preserving fiber integrity for composite applications [11].



**Figure 21.** Water absorption (WA, %) of okra (*Abelmoschus esculentus*) fibers obtained by different extraction methods. The left panel shows biological and combined extractions (raw, EB1–EB8), while the right panel presents chemical extraction at different NaOH concentrations (2.5%, 5%, and 7.5%). Values are expressed as mean  $\pm$  standard deviation.

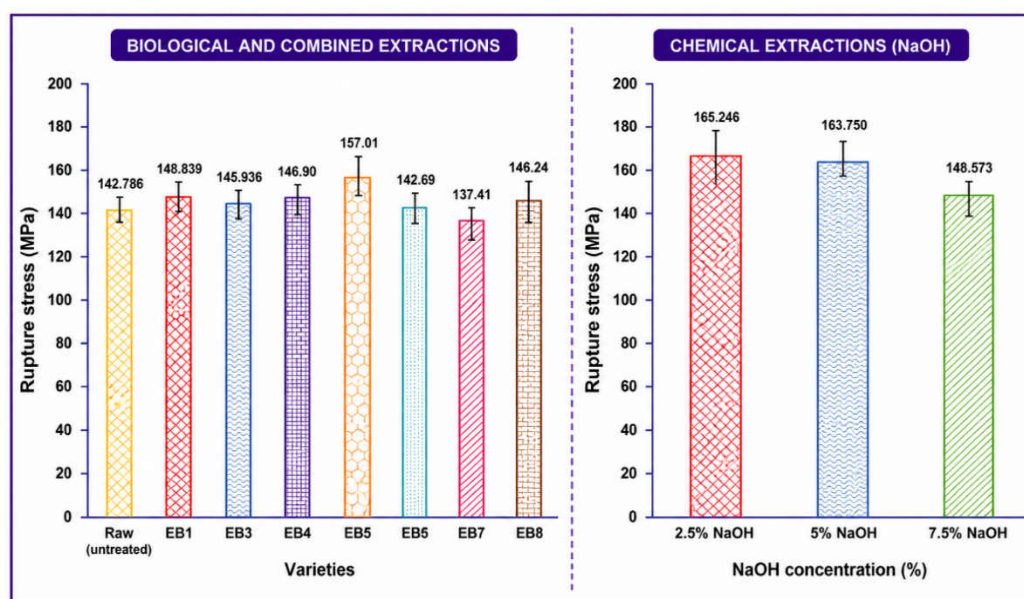


**Figure 22.** Young's modulus of okra (*Abelmoschus esculentus*) fibers obtained by different extraction methods. The left panel shows fibers extracted by retting (raw and treated samples 1–8), while the right panel presents

fibers extracted using different NaOH concentrations (2.5%, 5%, and 7.5%). Values are expressed as mean  $\pm$  standard deviation.

### 3.5.2. Rupture Stress

As shown in **Figure 23**, the rupture stress of okra (*Abelmoschus esculentus*) fibers is significantly influenced by the extraction method and treatment conditions. For biological (retting) extraction, the rupture stress shows moderate variations between raw and treated fibers (EB1–EB5), followed by a noticeable increase for EB6–EB8. This behavior can be attributed to the gradual removal of pectins and surface impurities, which improves fiber individualization and enhances stress transfer along the cellulose microfibrils [5]. Similar trends have been reported for natural fibers undergoing retting, where controlled degradation of non-cellulosic materials leads to improved mechanical performance [17]. For chemical extraction, rupture stress increases markedly with NaOH concentration (2.5% to 7.5%). This improvement is associated with the removal of lignin and hemicellulose, resulting in better alignment of cellulose chains and increased crystallinity [12]. Such effects have been widely reported for alkali-treated fibers, where mechanical strength is enhanced due to improved interfacial bonding and reduced amorphous content [13,21]. At higher concentration (7.5%), the highest rupture stress is obtained, indicating efficient purification and improved load-bearing capacity of the fibers. However, excessive alkali treatment may induce microstructural damage or cellulose degradation, potentially reducing strength beyond optimal conditions [24]. Overall, both extraction methods enhance rupture stress, with chemical treatment showing a more pronounced effect due to more effective removal of non-cellulosic components. These results are consistent with previous studies highlighting the importance of fiber purification and microstructural organization in determining mechanical performance [11,19].

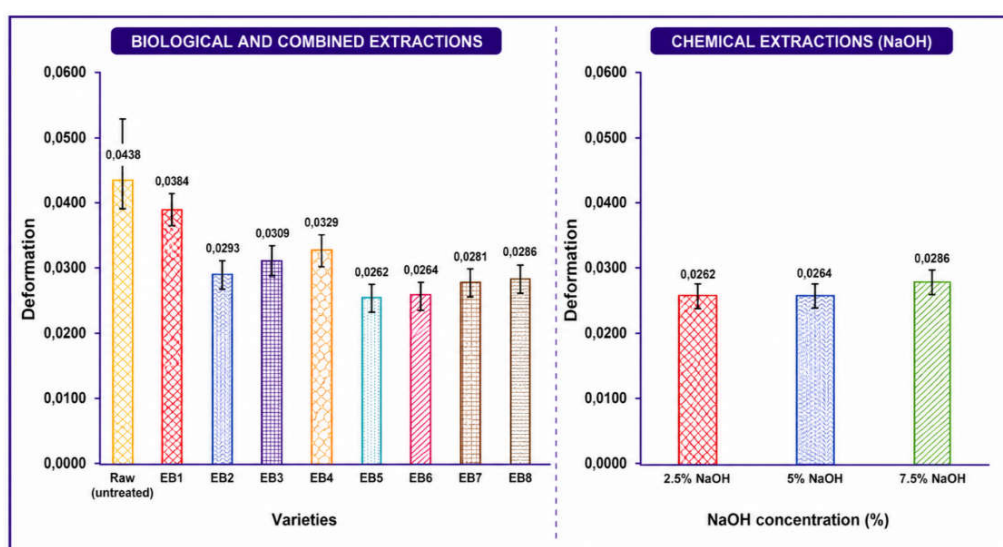


**Figure 23.** Rupture stress of okra (*Abelmoschus esculentus*) fibers obtained by different extraction methods. The left panel shows fibers extracted by retting (raw and treated samples 1–8), while the right panel presents fibers extracted using different NaOH concentrations (2.5%, 5%, and 7.5%). Values are expressed as mean  $\pm$  standard deviation.

### 3.5.3. Deformation

As shown in **Figure 24**, the deformation (strain) of okra (*Abelmoschus esculentus*) fibers is strongly dependent on the extraction method and treatment conditions. For **biological and combined extractions** (left panel), raw fibers exhibit the highest deformation due to the presence of amorphous constituents such as hemicellulose, lignin, and pectins, which impart flexibility to the fiber structure

[5]. As treatment progresses (EB1–EB8), a general decrease in deformation is observed, indicating the progressive removal of these non-cellulosic components and improved fiber individualization [10,15]. This reduction in strain is consistent with enhanced microfibril alignment and increased stiffness, as reported for retted natural fibers [17]. For **chemical extraction** (right panel), deformation values remain relatively low and slightly increase with NaOH concentration. The alkaline treatment removes hemicellulose and lignin, leading to a more crystalline cellulose structure and reduced ductility [12]. Similar behavior has been widely reported, where alkali-treated fibers exhibit lower elongation due to increased rigidity and reduced amorphous content [13,21]. At higher NaOH concentration (7.5%), a slight increase in deformation may be observed, which can be attributed to partial structural damage or surface fibrillation induced by excessive chemical treatment [24]. Such effects may locally weaken the fiber structure despite the overall increase in cellulose content. Overall, both extraction methods reduce fiber deformability, with chemical treatment producing stiffer and less extensible fibers. The balance between stiffness and ductility is critical for composite applications, where optimized mechanical behavior is required [11,19].



**Figure 24.** Deformation (strain) of okra (*Abelmoschus esculentus*) fibers obtained by different extraction methods. The left panel shows fibers extracted by retting (raw and treated samples 1–8), while the right panel presents fibers extracted using different NaOH concentrations (2.5%, 5%, and 7.5%). Values are expressed as mean  $\pm$  standard deviation.

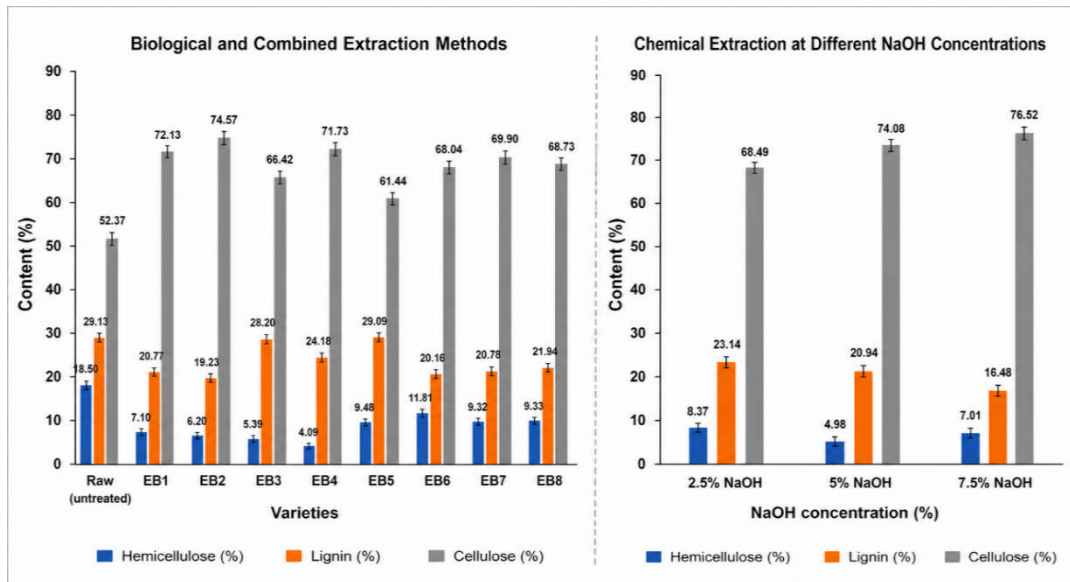
#### 3.5.4. Chemical Composition

As shown in **Figure 25**, the chemical composition of okra (*Abelmoschus esculentus*) fibers is significantly influenced by the extraction method and NaOH concentration. For biological and combined extractions, a clear increase in cellulose content is observed compared to raw fibers, accompanied by a reduction in hemicellulose and partial removal of lignin. This behavior is attributed to the progressive elimination of amorphous components such as hemicellulose and surface impurities, leading to a more cellulose-rich structure [5]. Similar trends have been reported for natural fibers subjected to retting or combined treatments, where selective removal of matrix components improves fiber purity [10,15].

For chemical extraction, increasing NaOH concentration (2.5% to 7.5%) results in a marked decrease in lignin and hemicellulose contents, while cellulose content increases significantly. This confirms that alkaline treatment effectively disrupts lignin–carbohydrate complexes and removes non-cellulosic materials [12]. Such behavior is widely documented for lignocellulosic fibers, where NaOH treatment enhances cellulose exposure and fiber crystallinity [13,21]. At higher concentration (7.5%), the highest cellulose content is obtained, indicating efficient delignification and purification

of the fibers. However, excessive chemical treatment may also induce partial degradation of cellulose chains or structural alteration [24], which can affect mechanical performance [19].

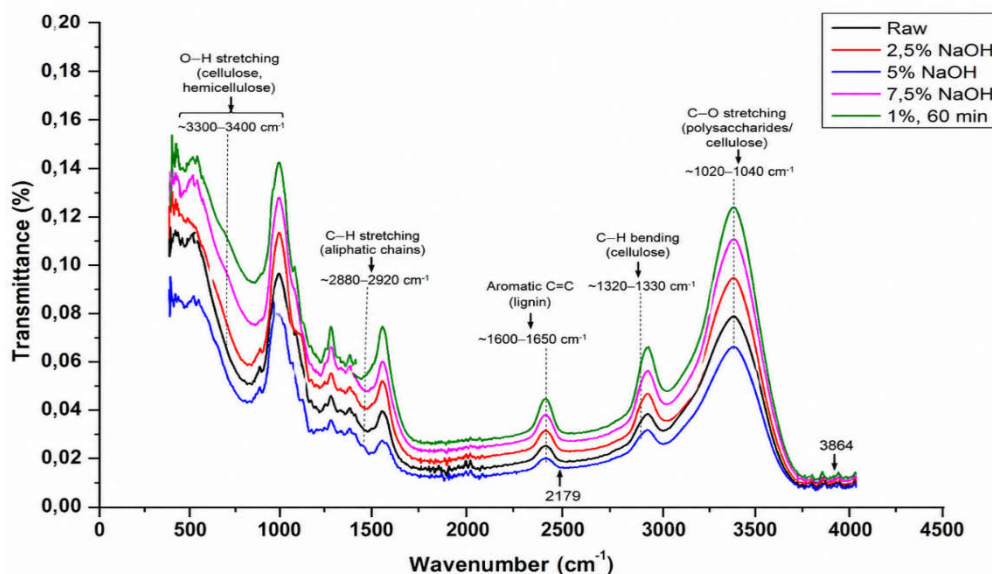
Overall, both extraction methods improve fiber chemical composition by increasing cellulose content, but chemical treatment shows a stronger effect. The combined method provides a more moderate modification, preserving fiber integrity while enhancing composition, which is advantageous for composite applications [11].



**Figure 25.** Chemical composition (%) of *Abelmoschus esculentus* fibers, including cellulose, hemicellulose, and lignin contents, as a function of extraction method and NaOH concentration.

### 3.6. FTIR

The FTIR spectra of okra (*Abelmoschus esculentus*) fibers under different extraction conditions are presented in **Figure 26**.



**Figure 26.** FTIR spectra of okra (*Abelmoschus esculentus*) fibers under different extraction conditions. The spectra show characteristic absorption bands associated with cellulose, hemicellulose, and lignin. Changes in peak intensity and position after biological and chemical treatments indicate the progressive removal of non-cellulosic components and the enrichment of cellulose in the fiber structure.

The main absorption bands identified in the FTIR spectra and their corresponding functional groups are presented in Table 1.

**Table 1.** Main FTIR absorption bands identified in okra (*Abelmoschus esculentus*) fibers and their corresponding functional groups.

Wavenumber (cm <sup>-1</sup> )	Functional Group
3330–3400	O–H stretching
2920	C–H stretching
1730	C=O hemicellulose
1600–1510	Aromatic lignin
1240	C–O lignin
1030	C–O cellulose

The spectra exhibit characteristic peaks of lignocellulosic materials, confirming the presence of cellulose, hemicellulose, and lignin. The broad band observed around ~3300–3400 cm<sup>-1</sup> corresponds to O–H stretching vibrations, associated with hydroxyl groups in cellulose and hemicellulose. A noticeable increase in intensity for treated fibers indicates enhanced exposure of hydroxyl groups due to the removal of surface impurities and amorphous components [12]. The peaks located at ~2880–2920 cm<sup>-1</sup> are attributed to C–H stretching vibrations of aliphatic chains. A slight reduction in intensity after chemical treatment suggests partial removal of waxes and non-cellulosic materials [13]. The absorption bands observed in the region ~1600–1650 cm<sup>-1</sup> are associated with lignin aromatic structures. The decrease in these peaks for NaOH-treated fibers confirms the removal of lignin during alkaline treatment [21]. The peak around ~1320–1330 cm<sup>-1</sup> corresponds to C–H bending and O–H deformation of cellulose. The relative increase in this peak intensity after treatment indicates enrichment in cellulose content [5]. In addition, the band near ~1020–1040 cm<sup>-1</sup> is attributed to C–O stretching vibrations in polysaccharides. The enhancement of this peak further confirms the purification of cellulose following chemical extraction [12]. Overall, the FTIR results demonstrate that alkaline treatment effectively removes hemicellulose and lignin, leading to a cellulose-rich structure. The sample treated at higher NaOH concentration shows the most pronounced changes, indicating a more efficient delignification process. However, excessive treatment may also alter the fiber structure, as suggested by peak modifications at higher wavenumbers.

### 3.7. DRX

The X-ray diffraction patterns of okra (*Abelmoschus esculentus*) fibers obtained under different extraction conditions are presented in **Figure 27**. All samples exhibit typical diffraction peaks of cellulose I, confirming the semicrystalline nature of lignocellulosic fibers. The main reflections are observed at  $2\theta \approx 14.8^\circ$  (–110),  $16.5^\circ$  (110),  $22.6^\circ$  (200), and  $34.5^\circ$  (004), which are characteristic of native cellulose structure [28,34]. The intensity of the dominant peak at  $2\theta \approx 22.6^\circ$  (200) varies significantly with extraction conditions, indicating changes in the degree of crystallinity. The crystallinity index (CrI) was estimated using the Segal method according to Equation (18)

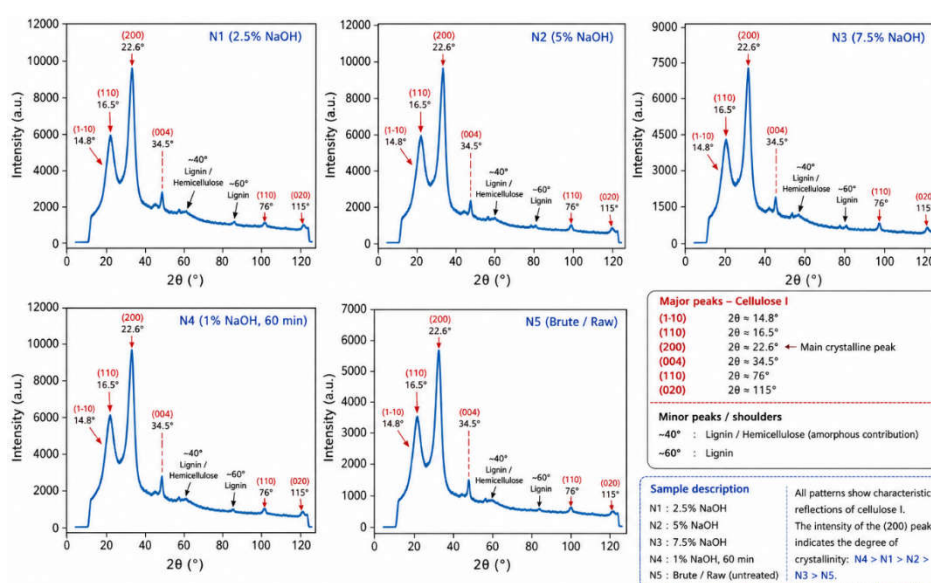
$$CrI(\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (18)$$

where:

- $I_{200}$  is the maximum intensity of the crystalline peak at  $2\theta \approx 22^\circ$ ,
- $I_{am}$  is the intensity of the amorphous region at  $2\theta \approx 18^\circ$ .

Chemically treated fibers (N1–N3) show a higher peak intensity compared to raw fibers (N5), suggesting an increase in crystallinity due to the removal of amorphous constituents such as hemicellulose and lignin during alkaline treatment [12,13]. This behavior is consistent with previous studies reporting that alkali treatment enhances cellulose crystallinity by eliminating disordered

regions [21,24]. Among the treated samples, the fiber extracted at moderate NaOH concentration (N2: 5%) exhibits a well-defined and sharp (200) peak, indicating an optimal balance between purification and structural preservation. At higher concentration (N3: 7.5%), although the crystallinity remains high, slight peak broadening may be observed, suggesting partial disruption of the crystalline domains due to excessive chemical attack [13]. The sample obtained under combined treatment (N4: 1% NaOH, 60 min) also shows improved peak intensity compared to raw fibers, confirming that even mild alkaline conditions contribute to partial removal of amorphous materials while preserving fiber integrity [10,18]. Additionally, low-intensity and broad features observed around  $\sim 40^\circ$  and  $\sim 60^\circ$  are associated with residual amorphous phases, mainly lignin and hemicellulose [5]. The reduction of these contributions in treated fibers further confirms the effectiveness of chemical extraction. Overall, the XRD results demonstrate that alkaline treatment enhances the crystalline structure of okra fibers by removing non-cellulosic components. This increase in crystallinity is directly correlated with improved mechanical properties, as higher crystalline content generally leads to increased stiffness and strength of natural fibers [11,28,30].



**Figure 27.** X-ray diffraction (XRD) patterns of okra (*Abelmoschus esculentus*) fibers subjected to different extraction conditions. Samples include N1 (2.5% NaOH), N2 (5% NaOH), N3 (7.5% NaOH), N4 (1% NaOH, 60 min), and N5 (raw fibers). All patterns exhibit characteristic diffraction peaks of cellulose I, notably at  $2\theta \approx 14.8^\circ$  ( $-110$ ),  $16.5^\circ$  (110),  $22.6^\circ$  (200), and  $34.5^\circ$  (004). The intensity of the main peak at  $2\theta \approx 22.6^\circ$  reflects the degree of crystallinity, which increases after chemical treatment. Minor peaks around  $\sim 40^\circ$  and  $\sim 60^\circ$  are associated with amorphous components such as lignin and hemicellulose.

#### 4. Discussion

The present study was based on the hypothesis that controlled alkaline treatment, alone or combined with biological retting, would improve the physicochemical and mechanical properties of *Abelmoschus esculentus* fibers through the removal of non-cellulosic components while preserving cellulose integrity. The results obtained confirm this hypothesis.

The progressive decrease in hemicellulose and lignin content, accompanied by an increase in cellulose fraction, demonstrates the effectiveness of alkaline treatment in modifying fiber composition. The observed improvement may result from the progressive removal of amorphous constituents and improved cellulose organization [12,13]. Similar trends have been observed in other natural fibers such as kenaf and pineapple, where chemical treatment improves fiber purity and performance [10,15].

FTIR analysis confirms these chemical modifications, with a reduction in lignin-related bands ( $\sim 1600\text{--}1650\text{ cm}^{-1}$ ) and an increase in cellulose-associated peaks ( $\sim 1020\text{--}1040\text{ cm}^{-1}$ ), indicating cellulose enrichment and improved structural organization [21,24,33]. XRD results further support these findings by showing an increase in crystallinity after treatment, particularly through the enhancement of the diffraction peak at  $2\theta \approx 22.6^\circ$ , which is characteristic of cellulose I [28,30]. However, at higher NaOH concentrations, slight peak broadening suggests partial degradation of crystalline domains, indicating that excessive treatment may negatively affect fiber structure [24].

The evolution of physical properties reflects these structural changes. The increase in fiber density is associated with the removal of low-density amorphous components and the formation of a more compact structure [26,29]. Similarly, the improvement in fiber fineness results from the progressive separation of fiber bundles into elementary fibers due to the weakening of inter-fibrillar bonds [12]. In contrast, the decrease in Moisture regain and water absorption with increasing NaOH concentration is attributed to the reduction of hydrophilic groups, particularly hemicellulose, which plays a key role in water uptake [14,19]. This reduction in hygroscopicity is advantageous for applications requiring dimensional stability.

Mechanical properties further confirm the beneficial effect of controlled treatment. The increase in Young's modulus and tensile strength is attributed to improved cellulose crystallinity and better alignment of microfibrils, which enhance stress transfer along the fiber axis [28,30]. These results are consistent with previous studies showing that alkaline treatment improves the stiffness and strength of natural fibers [12,13,21]. Conversely, the decrease in deformation reflects reduced ductility due to the removal of amorphous components that contribute to fiber flexibility [19]. At higher NaOH concentrations, slight variations in mechanical behavior may be attributed to structural damage or cellulose degradation caused by excessive chemical treatment [24].

In a broader context, these findings demonstrate that the performance of okra fibers is governed by a balance between purification and structural preservation. While alkaline treatment significantly enhances fiber properties, excessive treatment may lead to degradation of the cellulose structure. The combined extraction method appears to offer the most suitable compromise, ensuring sufficient removal of non-cellulosic components while maintaining fiber integrity. The optimal condition identified (1 wt% NaOH, 60 min) confirms that moderate treatment is sufficient to achieve significant improvements without inducing degradation, in agreement with optimization strategies reported in the literature [23,25].

From an application perspective, the improved mechanical performance, increased cellulose content, and reduced hygroscopicity indicate that these fibers are promising candidates for reinforcement in polymer composites. Their relatively low density and enhanced stiffness make them suitable for lightweight and sustainable materials, contributing to the development of bio-based composites [1,3,11].

Future work should focus on optimizing extraction parameters using advanced statistical approaches, evaluating fiber-matrix interfacial adhesion in composite systems, and investigating long-term durability under environmental conditions. In addition, hybridization with other natural or synthetic fibers could be explored to further enhance performance.

## 5. Conclusions

This study investigated the influence of biological, chemical, and combined extraction methods on the physicochemical, mechanical, and structural properties of *Abelmoschus esculentus* fibers. The results demonstrate that extraction conditions significantly affect fiber performance through the progressive removal of non-cellulosic components such as hemicellulose and lignin.

Alkaline treatment led to an increase in cellulose content and crystallinity, as confirmed by FTIR and XRD analyses, resulting in improved mechanical properties, particularly Young's modulus and tensile strength. At the same time, a reduction in moisture regain and water absorption was observed, indicating improved dimensional stability. However, excessive alkaline treatment was found to induce partial degradation of the fiber structure, leading to a decrease in performance.

Among the tested conditions, the combined extraction method provided the best compromise between fiber purification and structural preservation. The optimal condition (1 wt% NaOH, 60 min) resulted in fibers with enhanced mechanical performance, reduced hygroscopicity, and improved structural organization. These findings demonstrate that okra fibers extracted under optimized alkaline conditions can serve as sustainable lightweight reinforcements for advanced bio-composite applications.

Overall, the results confirm that okra (*Abelmoschus esculentus*) fibers are promising candidates for sustainable composite applications. Their low density, improved stiffness, and reduced water sensitivity make them suitable for use as reinforcement in lightweight bio-based materials.

**Supplementary Materials:** The following supporting information can be downloaded at: Preprints.org. The supplementary materials include all statistical analyses and graphical representations related to the studied properties, including: Complete experimental design; Factorial regression coefficients; Analysis of variance (ANOVA) tables; Alias structures; Factorial plots; Contour plots; Response surface plots. The investigated properties are as follows: Water absorption rate (WA%) ; Tensile strength; Elongation at break; Young's modulus; Hemicellulose content; Cellulose content; Lignin content; Fineness; Moisture regain; Density.

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